Clean Water Act Section 319(h) Nonpoint Source Pollution Control Program Project

Phytoremediation of Excessively High Phosphorus Soils and Subsequent Reduced Phosphorus Runoff into the North Bosque River

TSSWCB Project #FY04-10

(**Federal ID No.** C9-996236-11)

Quality Assurance Project Plan

Revision #1

Texas State Soil and Water Conservation Board

prepared by:

Texas Agricultural Experiment Station
and
Tarleton State University

Effective Period: Upon EPA Approval for 36 months

Questions concerning this quality assurance project plan should be directed to:

Dr. James P. Muir
1229 North U.S. Hwy 281
Texas Agricultural Experiment Station
Stephenville, Texas 76402
(254) 968-4144
j-muir@tamu.edu

A1 APPROVAL PAGE

Environmental Protection Agency

5/3/07	5/3/07
Ellen Caldwell — Date EPA Project Officer State/Tribal Programs Branch	Donna Miller — Date Chief State/Tribal Programs Branch

Texas State Soil and Water Conservation Board

4-4-07	4/4/07
T. J. Helton — Date Project Manager	Donna Long — Date QAO

Texas Agricultural Experiment Station (TAES)

2/2/07	2/2/07
James Muir — Date TAES Project Manager/QAO/Data manager	Randy Bow — Date Field Manager

RICHARD WOLFE 2/2/07

Richard Wolfe — Date
Lab Manager

Texas Cooperative Extension (TCE)

2/23/07

Saqib Mukhtar — Date
Project Engineer

Tarleton State University (TSU)

2/6/07

David Weindorf — Date
TSU Soil Scientist

2-6-07

Roger Wittie — Date
TSU Plant Scientist

Texas Institute for Applied Environmental Research (TIAER)

2/16/07

Mark Murphy — Date
Laboratory Manager

A2 TABLE OF CONTENTS

LIST OF ACRONYMS

BMP	**Best Management Practices**
CAR	**Corrective Action Report**
COC	**Chain-of Custody**
CNMP	**Comprehensive Nutrient Management Plan**
DOC	**Demonstration of Capability**
DP	**Dissolved orthophosphates**
DQO	**Data Quality Objective**
EOF	**Edge-of-field**
EP	**Extractible Phosphorus**
EPA	**Environmental Protection Agency**
FTP	**Forage Total Phosphorus**
FY	**Fiscal Year**
GPS	**Global Positioning System**
LCS	**Laboratory Control Standard**
LMU	**Land Management Unit**
MDMA	**Monitoring Data Management & Analysis**
NCR	**Non-conformance report**
NPS	**Nonpoint Source**
QA	**Quality Assurance**
QAM	**Quality Assurance Manual**
QAO	**Quality Assurance Officer**
QAPP	**Quality Assurance Project Plan**
QAS	**Quality Assurance Specialist**
QC	**Quality Control**
QMP	**Quality Management Plan**
RPD	**Relative Percent Deviation**
RL	**Reporting Limit**
SOP	**Standard Operating Procedure**
SWQM	**Surface Water Quality Monitoring**
TAES	**Texas Agricultural Experiment Station**
TIAER	**Texas Institute for Applied Environmental Research**
TCE	**Texas Cooperative Extension**
TCEQ	**Texas Commission on Environmental Quality**
TKN	**Total Kjeldahl Nitrogen**
TMDL	**Total Maximum Daily Load**
TN	**Total Nitrogen**
TP	**Total Phosphorus**
TS	**Total solids**
TSS	**Total Suspended Solids**
TSSWCB	**Texas State Soil and Water Conservation Board**
TSU	**Tarleton State University**
TSWQS	**Texas Surface Water Quality Standards**

A3 DISTRIBUTION LIST

Environmental Protection Agency Region 6
1445 Ross Avenue, Suite 1200
Dallas, Texas 75202-2733

Name: Ellen Caldwell (214) 665-7107
Title: EPA Project Officer

Texas Agricultural Experiment Station
2119 North U.S. Hwy 281
Stephenville, TX 76401

Name: Jim Muir, (254) 968-4144
Title: Project Manager/QAO

Name: Richard Wolfe
Title: TAES Lab Manager

Name: John R. Bow
Title: TAES Field Manager

Texas State Soil and Water Conservation Board
P.O. Box 658
Temple, Texas 76503

Name: T. J. Helton (254) 773-2250 ext. 234
Title: Project Manager

Name: Donna Long (254) 773-2250 ext. 228
Title: QAO

Texas Cooperative Extension (TCE)
303-B Scoates Hall, 2117
TAMU, College Station. Texas 77843-2117

Name: Saqib Muktar (979) 458-1019
Title: Project Manager

Tarleton State University (TSU)
Box T-0050
Stephenville, TX 76402

Name: David Weindorf (254) 968-9298
Title: TSU Soil Scientist

Name: Roger Wittie (254) 968-9221
Title: TSU Range Scientist

Texas Institute for Applied Environmental Research
Tarleton State University
Box T-0410
Stephenville, TX 76402

Name: Mark Murphy (254) 968-9570
Title: TIAER Laboratory Manager

A4 PROJECT/TASK ORGANIZATION

The following is a list of individuals and organizations participating in the project with their specific roles and responsibilities:

USEPA—United States Environmental Protection Agency, Region VI, Dallas. Provides project overview at the Federal level.

Ellen Caldwell, USEPA Texas Nonpoint Source Project Officer

Responsible for overall performance and direction of the project at the Federal level. Ensures that the project assists in achieving the goals of the federal Clean Water Act (CWA). Reviews and approves the quality assurance project plan (QAPP), project progress, and deliverables.

TSSWCB—Texas State Soil and Water Conservation Board, Temple, Texas. Provides project overview at the State level.

Thomas J. Helton, TSSWCB Project Manager

Responsible for ensuring that the project delivers data of known quality, quantity, and type on schedule to achieve project objectives. Tracks and reviews deliverables to ensure that tasks in the work plan are completed as specified.

Donna Long, TSSWCB Quality Assurance Officer

Determines that the project meets the requirements for planning, quality assurance (QA), quality control (QC), and reporting under the CWA Section 319 program. Reviews and approves QAPP and any amendments or revisions and ensures distribution of approved/revised QAPPs to TSSWCB and USEPA participants. Responsible for verifying that the QAPP is followed by project participants. Monitors implementation of corrective actions. Coordinates or conducts audits of field and laboratory systems and procedures.

TAES-Stephenville—Texas Agricultural Experiment Station, Stephenville, Texas. Project Lead.

James Muir, TAES Project Manager/QAO/Data Manager

Responsible for implementing and monitoring TSSWCB requirements in contracts, QAPP, and QAPP amendments and appendices. Ensures monitoring systems audits are conducted to ensure QAPP is followed by TAES participants and that projects are producing data of known quality. Ensures that subcontractors are qualified to perform contracted work. Ensures TSSWCB project managers and/or QA officers are notified of deficiencies and nonconformance, and that issues are resolved. Responsible for managing data, validating that data, and ensuring that data collected are acceptable for reporting to TSSWCB. Responsible for supervising sampling and oversight of project activities. Responsible for field scheduling, staffing, and ensuring that staff are appropriately trained. Responsible for compiling all reports prior to submission to TSSWCB.

Randy Bow, TAES Project Field Operations Supervisor

Responsible for supervising all field project activities which involve sampling and oversight of field phytoremediation and edge-of-field (EOF) monitoring related activities. Responsible for the acquisition of feed, manure, compost, soil, plant, and water samples and field data measurements in a timely manner that meet the quality objectives specified in Section A7 (table A.1) as well as the requirements of Sections B1 through B8. Reports status, problems, and progress to TAES project manager. .

Richard Wolfe, TAES Laboratory Manager

Responsible for supervision of TAES laboratory personnel involved in generating analytical data for the project. Responsible for ensuring that laboratory personnel involved in generating analytical data are adequately trained, have a thorough knowledge of the QAPP, and follow all SOPs specific to the analyses / task performed. Responsible for oversight of all TAES laboratory operations. Ensures that all QA/QC requirements are met, all laboratory documentation is complete, adequately maintained, and results are reported accurately. Enforces corrective action, as required. Monitors implementation of the laboratory quality assurance protocols to ensure compliance with project data quality objectives as defined by the QAPP. Responsible for supervising and verifying all aspects of QA/QC in the laboratory. Ensures that all QA reviews and audits are conducted in a timely manner.

TCE—Texas Cooperative Extension, College Station, Texas. Provides engineering support in the form of project demonstration design and installation.

Saqib Mukhtar, TCE Project Engineer

Responsible for designing and installing EOF surface water runoff equipment; responsible for collecting and analyzing EOF runoff samples from forage and buffer strips on all project sites. He will collect these data, analyze them statistically and report to the TAES project manager all findings. He will also participate in making these findings available for publication, extension and education purposes. Responsible for providing the project manager with all reportable activities.

To be hired, TCE Forage Specialist

Responsible for designing and monitoring cultivated forage phytoremediation and demonstration on the dairies; responsible for planting, harvesting and analyzing forage samples on high-P manured soils on dairies, both functional and abandoned. She will collect data, analyze and report these to the TAES project manager as well as provide these to dairy producers in the region. Responsible for providing the project manager with all reportable activities.

TSU—Tarleton State University, Stephenville, Texas. Provides field, laboratory, and analytical support for the project.

David Weindorf, TSU Soil Scientist

Responsible for collecting and analyzing soil samples from dairies, forage plots, buffer strips, and manure/compost material. Determines mineral analyses, conducts statistical analyses and provides all reportable findings to the TAES project manager. Participates in making these findings available for publication, extension and education purposes.

Roger Wittie, TSU Rangeland Scientist

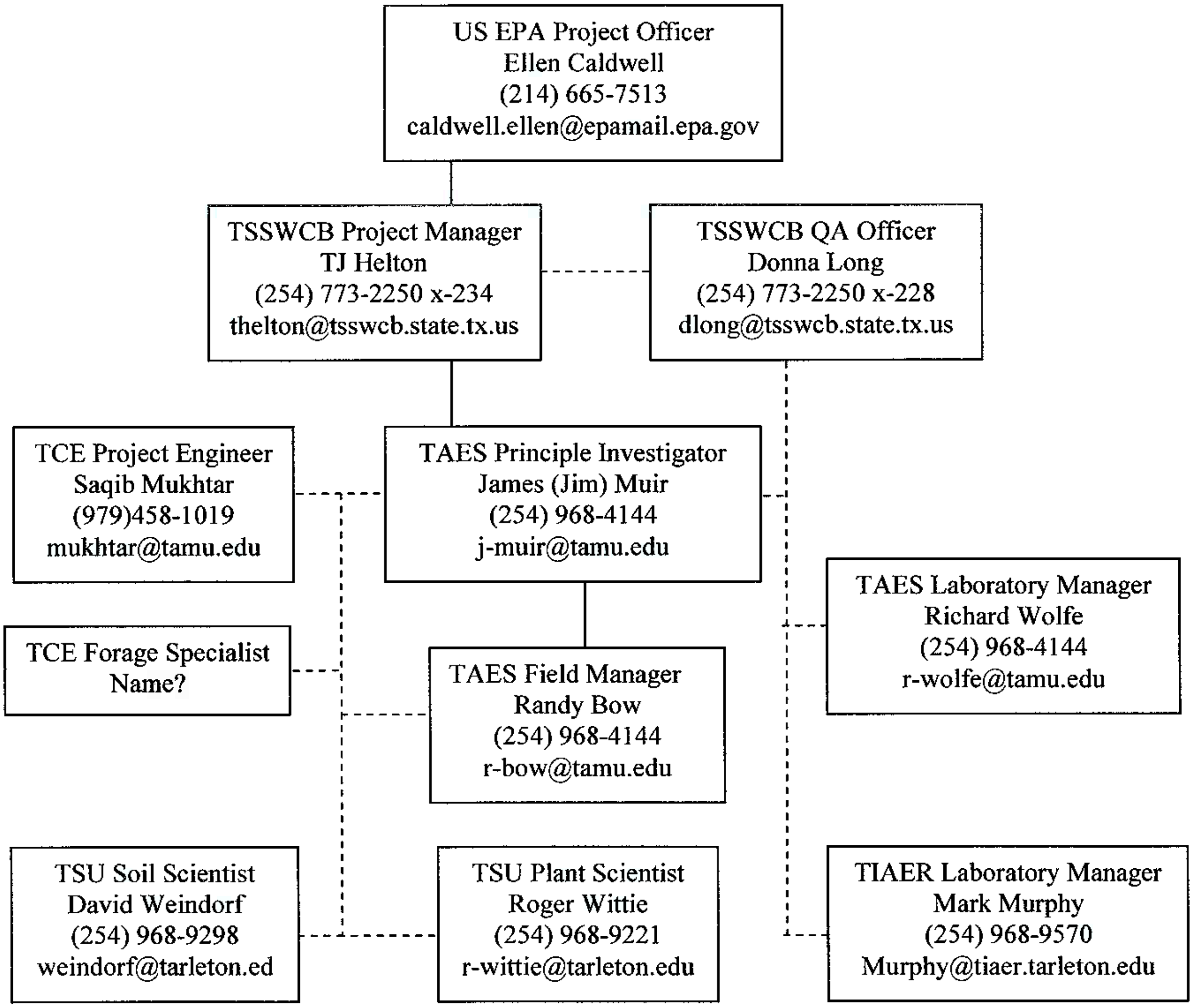
Responsible for design and establishment of vegetative buffer strips at all locations, collection of data from the vegetation, statistical analyses, and interpretion of findings. Provides all reportable findings to the TAES project manager . Participates in making these findings available for publication, extension and education purposes.

TIAER—Texas Institute for Applied Environmental Research, Stephenville, Texas. Provides laboratory analytical support.

Mark Murphy,TIAER Laboratory Manager

Responsible for supervision of TIAER chemistry laboratory personnel involved in generating analytical data for this project. Responsible for ensuring that laboratory personnel involved in generating analytical data are adequately trained, have a thorough knowledge of the QAPP, and follow all standard operating procedures (SOPs) specific to the analysis / task performed. Responsible for oversight of all TIAER laboratory operations and ensuring that all QA/QC requirements are met. Responsible for documentation related to laboratory analyses. Responsible for providing TCE EOF monitor with data and for managing/storing that data according to QAPP directives. Enforces corrective action, as required. Develops and facilitates laboratory system audits with TIAER QA officer.

Figure A4-1. Organization flow chart.



A5 PROBLEM DEFINITION/BACKGROUND

The purpose of this QAPP is to clearly delineate TAES/TSU/TCE QA policy, management structure, and procedures which will be used to implement the QA requirements necessary to verify and validate the forage, soil, and water data collected. The QAPP is reviewed by the TSSWCB to help ensure that data generated for the purposes described above are scientifically valid and legally defensible. It will be signed by all participants who will then sign a letter to document adherence to the QAPP (Attachment 1).

The purpose of this project is to (1) Document/demonstrate a measurable rate of P removal via forages from crop fields with histories of excessive dairy waste application. (2) Develop and test easily established vegetative buffer strips that harvest or stabilize soil-P in surface water runoff moving from dairy waste application fields to streams, rivers or other drainages. (3) Based on results from the previous tasks, model and demonstrate forage systems, first tier and second tier buffers to accurately predict maximum soil-P removal and stabilization rates (and, consequently, maximum tolerable additional dairy waste-P) for the soils in the North Bosque. (4) Model the economics associated with implementation of the integrated approaches outlined above. TCEQ approved two TMDLs for phosphorus in the North Bosque River Segments 1226 and 1255 on February 9, 2001, which were subsequently submitted to and approved by the United States Environmental Protection Agency (USEPA). The Implementation Plan for the two North Bosque River segments was approved by TCEQ in late 2002 and the Texas State Soil & Water Conservation Board (TSSWCB) in early 2003 (TCEQ and TSSWCB, 2002). This project will assess the effectiveness of using year-round forage systems and perennial vegetation buffer strips as potential solutions for nonpoint source related phosphorus reduction measures in the North Bosque River watershed. Additional specifications of the problem to be addressed under this QAPP are described in Appendix A, the project workplan.

Agencies cooperating in this project include TAES, TCE, and TSU. The TAES is responsible for coordination of the overall project and has subcontracted with TSU to perform soil and vegetation monitoring. TCE is responsible for EOF surface water quality monitoring. The TSSWCB has provided the funding for the plant, soil, and edge of field portion of this project. TAES/TCE/TSU will utilize its own laboratories for soil, forage, water (TIAER), and dairy manure/compost analysis.

A6 PROJECT/TASK DESCRIPTION

The main objective for this project is to reduce surface water contamination in the north Bosque River from soil-applied P of dairy manure origin.

Task 1. Document/demonstrate a measurable rate of P removal via forages from crop fields with histories of excessive dairy waste application.

- Subtask1.1. Locate three crop fields on the upper North Bosque River that have an excess of 200 ppm plant-available P due to historical applications of dairy wastes. Measure all components (both stable and plant-available) of P in the soil.
- Subtask 1.2: On these fields, determine optimal season length and P-extraction of cool season winter grasses and legumes on small plots by measuring both forage yields and P concentrations. Individual species will be tested years 1-2. Deliverables will include QAPP and reports of individual forage or grain crop P concentration.
- Subtask 1.3: On these same fields but distinct plots, determine optimal season length and P-extraction of warm season winter grasses and legumes on small plots by measuring both forage yields and P concentrations. Individual species will be tested years 1-2. Deliverables will include QAPP and reports of individual forage or grain crop P concentration.
- Subtask 1.4: Based on results from subtasks 1.2 and 1.3, design and demonstrate year-round forage production systems that have compatible growing seasons (do not overlap) and extract the maximum amount of plant-available P from these crop fields. Determine which systems extract the greatest amount of soil-P and determine if the plant-available P extraction affects stable P in the soils. These systems will be demonstrated in years 2 & 3. Deliverables will include QAPP and reports of forage systems P extraction (a function of forage P concentration and herbage yield) and the effects on soil P components (both total P and water soluble P fractions.

Task 2. Develop and test easily established vegetative buffer strips that harvest or stabilize soil-P in surface water runoff moving from dairy waste application fields to streams, rivers or other drainages.

- Subtask 2.1. Design and test first tier buffers composed of harvestable (hay) material with high soil-P extraction (yield X P concentration) potential that will catch and recycle surface runoff from croplands high in water-soluble P of dairy waste origin. Design and establish demonstrations (at least one site per soil type) will take place year 1; years 2-3 will involve soil, forage, and edge-of-field monitoring. Deliverables will include QAPP, buffer designs, and producer-oriented publications with buffer-establishment and management instructions.
- Subtask 2.2. Design and demonstrate second tier buffer strips for the specific soils, drainages and climate that will foster year-round buffering utilizing mostly (but not exclusively) native vegetation. These will consist of secondary tiers of mostly perennial species with ecologically stable environments. Designing and establishing demonstrations (at least one site per soil type) will take place year 1; years 2-3 will involve soil, forage, and edge-of-field monitoring. Deliverables will include QAPP, buffer designs, and producer-oriented publications with buffer-establishment and management instructions.

Task 3. Based on results from the previous tasks, model and demonstrate forage systems, first tier and second tier buffers to accurately predict maximum soil-P removal and stabilization rates (and, consequently, maximum tolerable additional dairy waste-P) for the soils in the North Bosque.

- Subtask 3.1. Develop, field test, and demonstrate to dairy producers and their neighbors currently accepting dairy wastes on their fields a crop field-to-stream model of how to maximize removal and stabilization of dairy waste P. This will entail utilizing results collected years 1 & 2 and will therefore be developed and field tested year 3. Deliverables will involve QAPP, theoretical models based on concrete experiences and, finally, producer-oriented publications that propose BMP's for bioremediation of high P soils.

Task 4. Model the economics associated with implementation of the integrated approaches outlined above. Data collection for this will take place from year 1-3 but final modeling will occur only during year 3. Deliverables will include producer-oriented documents for scenarios from Tasks 1-3 outlining costs and benefits of the BMP's being promulgated.

See Appendix A for the work plan tasks and schedule of deliverables for this project. This QAPP covers the soil, vegetation and EOF water runoff tasks of the work plan. No decisions will be made by the project team based on the data collected. These data will be subsequently analyzed and used by the TSSWCB. See Appendix B for sampling design and monitoring pertaining to this QAPP.

Amendments to the QAPP

Until the work described is completed, this QAPP shall be revised as necessary and reissued annually on the anniversary date, or revised and reissued within 120 days of significant changes, whichever is sooner. Requests for amendments are directed from the TAES Project Manager to the TSSWCB Project Manager in writing. They are effective immediately upon approval by the TSSWCB Project Manager and the EPA Project Officer. They will be distributed by the TAES Project Manager and incorporated into the QAPP by way of attachment and distributed to personnel on the distribution list.

Expedited Changes

Expedited Changes to the QAPP should be approved before implementation to reflect changes in project organization, tasks, schedules, objectives, and methods, address deficiencies and non-conformance, improve operational efficiency and accommodate unique or unanticipated circumstances. Requests for expedited changes are directed from the contractor TAES Project Manager to the TSSWCB Project Manager in writing. They are effective immediately upon approval by the TSSWCB Project Manager and QAO.

Expedited changes to the QAPP and the reasons for the changes shall be documented, and revised pages shall be initialed by the TAES and TSSWCB Project Managers and QAO, and the EPA Project Officer (if applicable), then distributed to all persons on the QAPP distribution list by the TAES Project Manager and EPA Project Officer. Expedited changes shall be reviewed, approved, and incorporated into a revised QAPP during the annual revision process or within 120 days of the initial approval in cases of significant changes.

A7 QUALITY OBJECTIVES AND CRITERIA

The measurement performance specifications to support the project objectives for a minimum data set are specified in Table A7.1 and in the text following. Note: Due to the high concentrations of solids and nutrients in EOF runoff samples, samples will not be filtered in the field.

Table A7.1 Measurement Performance Specifications.

PARAMETER	UNITS	METHOD	AWRL[1]	Laboratory Reporting Level (LRL)	PRECISION	BIAS	ACCURACY	LAB
WATER PARAMETERS								
Total Suspended Solids (TSS)	mg/L	EPA 160.2	4.0	4.0	20	80-120	80-120	TIAER
Ortho-Phosphate Phosphorus, dissolved, lab filtered (DP)	mg/L	EPA 365.2	0.04	0.005	20	80-120	80-120	TIAER
Total Solids (TS)	mg/L	SM 2540 B	10	10	20	80-120	80-120	TIAER
Conductivity	µS/cm	EPA 120.1	NA	NA	NA	NA	NA	TIAER
Total Kjeldahl Nitrogen (TKN)	mg/L	EPA 351.2, modified[2]	0.2	0.2	20	80-120	80-120	TIAER
Total Phosphorus (TP)	mg/L	EPA 365.4, modified[2]	0.06	0.06	20	80-120	80-120	TIAER
pH	pH units	EPA 150.1	NA	NA	NA	NA	NA	TIAER
SOIL PARAMETERS								
Extractable Phosphorus (EP)	mg/Kg	Mehlich III-ICP	NA	3.0	20	NA	NA	TAES
Total Nitrogen (TN)	mg/Kg	SSSA-Dumas Method	NA	0.01	20	NA	NA	TAES
Extractable Potassium	mg/Kg	Mehlich III-ICP	NA	3.0	20	NA	NA	TAES
Total Carbon	mg/Kg	SSSA-Dumas Method	NA	0.01	20	NA	NA	TAES
pH	pH units	EPA 9045 C	NA	NA	NA	NA	NA	TAES
FORAGE PARAMETERS								
Total Nitrogen (TN)	mg/Kg	SSSA-Dumas Method	NA	200	20	80-120	NA	TAES
Total Phosphorus(TFP)	mg/Kg	Feagley, et. al.	NA	200	20	80-120	NA	TAES
MANURE/COMPOST PARAMETERS								
Total Nitrogen (TN)	mg/Kg	SSSA-Dumas Method	NA	0.004	20	NA	NA	TAES
Total Phosphorus(TP)	mg/Kg	Mehlich III-ICP	NA	3.0	20	NA	NA	TAES

1 Precision criteria do not hold for values below laboratory RL. The same precision criteria apply to lab duplicate RPD.
2 Modification of the total phosphorus method involves using $CuSO_4$ instead of $HgSO_4$. Documentation of TIAER's ability to achieve acceptable performance using the modification is kept by the TIAER analytical laboratory. In case of equipment malfunction and resulting holding time issues, the alternate back-up analytical method for total phosphorus will be EPA 365.4, modified in the same way; EPA 351.1-4 for TKN; and lab-filtered EPA 365.2 (code 70507) for PO_4-P, EPA 365.4 for total P. EPA 351.2 for TKN states that the digestate may also be used for total P. If an alternative method is necessitated, all QC, AWRLs, recovery, precision, and bias limits required by TCEQ will be followed.

****References for soils, forage and manure/compost laboratory procedures can be found in the "References" section under "References for Table A7.1"**

Ambient Water Reporting Limits

The AWRLs specified in Table A7.1 are the program-defined reporting specifications for each analyte and yield data acceptable for routine water quality monitoring. The reporting limit is the lowest concentration at which the laboratory will report quantitative data within a specified recovery range. The laboratory will meet two requirements in order to report meaningful results to the TSSWCB:

- The laboratory's reporting limit for each analyte will be at or below the AWRL.
- The laboratory will demonstrate and document on an ongoing basis the laboratory's ability to quantify at its reporting limits.

Acceptance criteria are defined in Section B5.

Precision

Precision is a statistical measure of the variability of a measurement when a collection or an analysis is repeated and includes components of random error. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions.

Field splits are used to assess the variability of sample handling, preservation, and storage, as well as the analytical process, and are prepared by splitting samples in the field. Control limits for field splits are defined in Section B5.

Laboratory precision is assessed by comparing replicate analyses of laboratory control standards. Precision results are plotted on quality control charts, which are based on historical data and used during evaluation of analytical performance. Program-defined measurement performance specifications for laboratory control standard/laboratory control standard duplicate pairs are defined in Table A7.1.

Bias

Bias is a statistical measurement of correctness and includes multiple components of systematic error. A measurement is considered unbiased when the value reported does not differ from the true value. Bias is verified through the analysis of laboratory control standards prepared with certified reference materials and by calculating percent recovery. Results are plotted on quality control charts, which are calculated based on historical data and used during evaluation of analytical performance. Program-defined measurement performance specifications for laboratory control standards are specified in Table A7.1.

For soil samples, bias is verified through laboratory media standard results compared to replicated results of the same sample on a large volume basis. For vegetation, bias is verified through the analysis of laboratory media standards prepared with certified reference materials (NIST or NIST traceable standards). Therefore, bias will be determined in these analyses by comparing results of laboratory media standards to the historical average or to the certified reference results for NIST standards. Proper equipment calibration will serve to verify the bias for manure samples. Performance limits for bias are listed in Table A7.1.

Representativeness

Site selection, the appropriate sampling regime, the sampling of all pertinent media according to standard scientific SOPs, and use of only approved analytical methods will assure that the measurement data represents the conditions at the site.

Comparability

Confidence in the comparability of fixed/routine data sets for this project and for water quality assessments is based on the commitment of project staff to use only approved sampling and analysis methods and QA/QC protocols in accordance with quality system requirements and as described in this QAPP. Comparability is also guaranteed by reporting data in standard units, by using accepted rules for rounding figures, and by reporting data in a standard format as specified in Section B10.

Completeness

The completeness of the data is basically a relationship of how much of the data is available for use compared to the total potential data. Ideally, 100% of the data should be available. However, the possibility of unavailable data due to accidents, insufficient sample volume, broken or lost samples, etc. is to be expected. Therefore, it will be a general goal of the project(s) that 90% data completion is achieved.

A8 SPECIAL TRAINING/CERTIFICATION

New field personnel (Tarleton students) will receive training in proper sampling and field analysis. Before actual sampling or field analysis occurs, they will demonstrate to the designee appointed by the QA Officer their ability to properly calibrate field equipment and perform field sampling and analysis procedures. Training will be documented and retained in the personnel file and be available during a monitoring systems audit.

Laboratory analysts have a combination of experience, education, and training to demonstrate knowledge of their function. Laboratories have documented training records for each test that an analyst performs. Training is performed prior to analyzing samples and annually thereafter.

TAES & TSU personnel involved in use of Global Positioning System (GPS) for soil sampling locations have been trained in the appropriate use of GPS by David Weindorf who will follow the GPS Policy outlined in Attachment 2. No special certifications are required.

A9 DOCUMENTS AND RECORDS

The documents and records that describe, specify, report, or certify activities are listed in Table A9.1.

Data will be submitted to the TAES at the end of the project.

Table A9.1 Project Documents and Records

Document/Record	Location	Retention (yrs)	Format
QAPPs, amendments and appendices	TAES*	5 years	Paper
Field SOPs	TAES	5 years	Paper
Laboratory QA Manuals	TAES, TSU & TIAER Labs	5 years	Paper
Laboratory SOPs	TAES, TSU & TIAER Labs	5 years	Paper
QAPP distribution documentation	TAES, TSU & TIAER Labs	5 years	Paper
Field staff training records	TAES/TSU/TCE	5 years	Paper
Field equipment calibration/maintenance logs	TAES/TSU/TCE	5 years	Paper
Field instrument printouts	TAES/TSU/TCE	5 years	Paper
Field notebooks or data sheets	TAES/TSU/TCE	5 years	Paper/Electronic**
Chain of custody records	TAES/TSU/TCE/TIAER	5 years	Paper
Laboratory calibration records	TAES/TSU/TCE/TIAER	5 years	Paper/Electronic
Laboratory instrument printouts	TAES/TSU/TCE/TIAER	5 years	Paper/Electronic
Laboratory data reports/results	TAES/TSU/TCE/TIAER	5 years	Paper
Laboratory equipment maintenance logs	TAES/TSU/TCE/TIAER	5 years	Paper
Corrective Action Documentation	TAES	5 years	Paper

*TAES & TAES Lab are both in Stephenville, Texas.

**Electronic files are transferable to ASCII (DOS) pipe delimited text files.

Laboratory Data Reports

Data reports from the TAES, TSU and TIAER laboratories will report the test results clearly and accurately. The test report will include the information necessary for the interpretation and validation of data and will include the following:

- name and address of the laboratory
- name and address of the client
- a clear identification of the sample(s) analyzed
- identification of samples that did not meet QA requirements and why (e.g., holding times exceeded)
- date of sample receipt
- sample results

- field split results (as applicable)
- clearly identified subcontract laboratory results (as applicable)
- a name and title of person accepting responsibility for the report
- project-specific quality control results to include LCS sample results (% recovery), LCS duplicate results (%RPD), equipment, trip, and field blank results (as applicable), and RL confirmation (% recovery)
- narrative information on QC failures or deviations from requirements that may affect the quality of results.

In addition, a lab data report from the TAES (Stephenville) laboratory, with sample results and QC results, will be submitted to TAES for inclusion with project data submittals.

Electronic Data

Project data will be submitted electronically to TAES project manager in Excel files on both a diskette and as e-mail attachments (j-muir@tamu.edu) besides a hardcopy. The diskettes will be stored at TAES and files will be maintained on the Stephenville S (shared) drive. In addition, hardcopies will be stored at TAES. Individuals in A3 will be sent the most current copy of the QAPP by the TAES project manager.

B1 SAMPLING PROCESS DESIGN

Unless used for mapping purposes, all samples will be replicated by virtue of trial/demonstration designs in which at least 3 replications will be used. This approach is necessary for accurate statistical analyses. Specific water, soil, forage and manure/compost sampling process design is outlined in Appendix B.

B2 SAMPLING METHODS

Field Sampling Procedures

Field sampling will be conducted according to the sample handling procedures described in Section B3 and in Appendix B.

Sample volume, container types, minimum sample volume, preservation requirements, and holding time requirements are presented in Table B2.1.

Table B2.1 Sample Storage, Preservation and Handling Requirements

Parameter	Matrix	Container	Preservation	Sample Volume	Holding Time
Total Suspended Solids	water	HDPE	4° C	400 mL	7 days
pH	water	HDPE	4° C	150 mL	28 days
Conductivity	water	HDPE	4° C	150 mL	28 days
Total Solids	water	HDPE	4° C	150 mL	7 days
Ortho-Phosphorus, lab filtered	water	HDPE	4° C	150 mL	filter ASAP; 48 hrs until analysis
Total Kjeldahl Nitrogen	water	HDPE	4° C, pH<2 with H_2SO_4	200 mL	28 days
Total Phosphorus	water	HDPE	4° C, pH<2 with H_2SO_4	150 mL	28 days
Extractable Phosphorus	soil	plastic or glass	air dried	100 g	6 months
Extractable Potassium	soil	plastic or glass	air dried	100 g	6 months
Total Carbon	soil	Plastic or glass	air dried	100 g	6 months
pH	soil	Plastic or glass	air dried	100 g	6 months
Total Nitrogen	soil	plastic or glass	air dried	100 g	6 months
Total Nitrogen	forage	Plastic or glass	air dried	100 g	6 months
Total Phosphorus	forage	Plastic or glass	air dried	100 g	6 months
Total Nitrogen	manure	Plastic or glass	Air dried	100 g	6 months
Total Phosphorus	manure	Plastic or glass	Air dried	100 g	6 months

Sample Containers

The sample containers for most water samples are HDPE bottles. Clean plastic bags will be used to collect soil and manure samples. Plant samples will be collected in cloth bags, dried at 55° C, ground through a 1mm screen in a sheer mill, and stored in plastic/glass containers.

HDPE sample containers are cleaned for reuse by washing them in hot, soapy (non-phosphate) water. The bottles are rinsed first in warm tap water, then with 1 N HCl, and finally rinsed at least three times in type II ASTM water. They are placed on a rack to dry. Bags for soil, and manure samples are not reused but those for forage collection and drying are. The latter are turned inside-out, all plant particles are removed, turned back again and folded.

TIAER's tracking system (water samples) to detect any contamination resulting from the washing procedure is based on cleaning reference numbers. One method blank is evaluated for each batch of 20 samples. A sample bottle to be used for the method blank (identified as CB, for cleaning blank) is selected from the available clean bottles. Deionized water is poured into the CB sample bottle and it is treated just like the samples being analyzed that day. If any measured concentration is greater than the AWRL, corrective actions will be initiated. Sources of contamination are investigated and remediated, if found. Corrective action documentation is maintained for CB failures. Corrective actions include reanalyzing to confirm method blank contamination, investigating the source of the contamination, identifying all samples possibly affected by the contamination, and conferring with the TAES QAO & PM to determine if the data are acceptable.

Processes to Prevent Contamination

Samples will be collected directly into sample containers, when possible, to avoid contamination. Field QC samples (identified in Section B5) are collected to verify that contamination has not occurred.

Documentation of Field Sampling Activities

Field runoff sampling activities are documented on field data sheets, which are included in Appendix F. The following will be recorded for all samples:

1. Station ID
2. Location
3. Sampling time
4. Sampling date
5. Sampling depth, plot, species (as appropriate)
6. Sample collector's name/signature
7. Values for all measured field parameters (as appropriate)
8. Preservative added, if applicable
9. Detailed observational data (as appropriate), including:
    - water/soil/forage appearance
    - weather
10. Other observational data (as applicable), including:
    - activities in contributing fields that could impact samples (events impacting water quality, e.g., livestock watering upstream, etc.)

- unusual odors
- specific sample information (number of grabs, type, etc.)
- missing parameters (i.e., when a scheduled parameter or group of parameters is not collected)

Recording Data

For the purposes of this section and subsequent sections, all field and laboratory personnel follow the basic rules for recording information as documented below:

1. Legible writing in indelible ink with no modifications, write-overs or cross-outs;
2. Correction of errors with a single line followed by an initial and date;
3. Close-out on incomplete pages with an initialed and dated diagonal line.

Deficiencies, Non-conformance and Corrective Action Related to Sampling Requirements

Deficiencies are defined as unauthorized deviations from procedures documented in the QAPP or other applicable documents. Non-conformances are deficiencies that affect quality and render the data unacceptable or indeterminate. Deficiencies related to sampling methods requirements include, but are not limited to, such things as sample container, volume, and preservation variations, improper/inadequate storage temperature, holding-time exceeded, and sample site adjustments.

Deficiencies are documented in logbooks, field data sheets, etc. by field or laboratory staff and are reported to the cognizant field or laboratory supervisor via a corrective action report (CAR). The supervisor notifies the TAES Project Manager if the deficiency has the potential of being a nonconformance. The TAES Project Manager will notify the TSSWCB QAO of the potential nonconformance within 24 hours.

The TAES Project Manager, in consultation with TSSWCB QAO (and other affected individuals/ organizations), will determine if the deficiency constitutes a nonconformance. If it is determined the activity or item in question does not affect data quality and therefore, is not a valid nonconformance, the CAR will be completed accordingly and closed. If it is determined a nonconformance does exist, the TAES Project Manager in consultation with the TSSWCB QAO will determine the disposition of the nonconforming activity or item and necessary corrective action(s); results will be documented on the CAR.

CARs document root cause(s), impact(s), specific corrective action(s) to address the deficiency, action(s) to prevent recurrence, individual(s) responsible for each action, the timetable for completion of each action, and the means by which completion of each corrective action will be documented (see copy appendix E). CARs associated with non-conformances will be included with quarterly progress reports. In addition, significant conditions (i.e., situations which, if uncorrected, could have a serious effect on safety or on the validity or integrity of data) will be reported TSSWCB both verbally and in writing.

B3 SAMPLE HANDLING AND CUSTODY

Chain-of-Custody

Proper sample handling and custody procedures ensure the custody and integrity of samples beginning at the time of sampling and continuing through transport, sample receipt, preparation, and analysis.

Water quality data are generated in the field and the TAES/TSU/TIAER analytical laboratories. A chain of custody (COC) form is used to record sample identification parameters and to document the submission of samples from the field staff to the analytical laboratory staff. Each COC has space to record data for at least 15 separate samples. A copy of the COC is found in Appendix C. For grab samples, a field data sheet for each site is attached to the COC. COCs and accompanying data sheets are kept in three-ring binders in the TAES office for at least five years.

A sample is in custody if it is in actual physical possession or in a secured area that is restricted to authorized personnel. The COC form is used to document sample handling during transfer from the field to the laboratory and among subcontract laboratories. The following information concerning the sample is recorded on the COC form (See Appendix C). The list of included items should match the COC form in Appendix C. These are standard requirements for COC forms.

1. Date and time of collection
2. Site identification
3. Sample matrix
4. Number of containers, if applicable
5. Preservative, if applicable
6. Color code to indicate required analyses
7. Name of collector
8. Custody transfer signatures and dates and time of transfer
9. Bill of lading

Sample Labeling

Water samples are labeled on the container with an indelible marker. Label information includes:

1. Dairy, field and GPS identification
2. Time of sampling (or bottle number for composited samples)

These two unique identifiers can be matched with data on Chain of Custody forms when submitting samples. All samples are submitted on a daily basis and given a unique sample number. This sample identification number, time, and station location serve to match the sample with the data on the COC. All water samples are submitted to the laboratory on ice. Project samples do not require additional types of preservation prior to receipt by the laboratory. No samples for this project are field filtered.

Soil, forage, and manure sample labeling are described in the paragraph for each media type.

Sample Handling

Each sample container is labeled in the field with the identification stated above. Water samples are preserved on ice in a cooler while they are being transported to the laboratory. The field staff member documents in a field data sheet, COC form, or sample bench sheet the station, date, time, location, and sample type. A sample identification number is assigned to water samples at the TAES office and is written on the sample container and on the COC. The sample number, location, date, changes in possession and other pertinent data are recorded in ink on the COC, which accompanies all sets of sample containers. The field staff member transfers possession of the samples to a laboratory staff member or alerts a laboratory staff member and leaves the sample containers, COCs and other paperwork in a secured area. The field staff member and the laboratory staff member both sign and date the COC. Copies of the COC form used on this project are included as Appendix C.

Wet Weather (Storm) Samples. Field sub-plots will be isolated from overland flow to capture all the natural rainfall runoff resulting from up to a 25-yr, 24-h rainfall. From composite samples of captured runoff, laboratory analyses will be conducted to determine concentrations of Total Kjeldahl N (TKN), Total P (TP), Dissolved P (DP), Total Solids (TS), Total Suspended Solids (TSS), pH, and conductivity from each sample.

Soil Samples. Soil samples will be air-dried for at least 24 hours prior to shipping to the Stephenville TAES/TSU laboratories for analysis. Each soil sample will be placed in a soil sample bag, with sample identification marked on the outside of the sample bag. The label on the soil sample bag will contain the sample identification number (GPS reading or plot number), the dairy location, the LMU, and the depth(s) from which the sample was taken. An Excel spreadsheet will be completed for each day and site of sampling and printed in duplicate. One copy of the soil sample information sheet (Appendix H) will accompany the composite samples to the laboratory and one copy will be included in the project file at TAES/TSU.

Forage Samples. Each forage sample will be placed in a cloth bag, sealed, and marked with sample identification on a tag on the outside of the bag. The sample identification will identify the dairy and the demonstration/plot from which the sample was taken. Bags containing forage samples will be transported to TAES Stephenville. A "Forage Sample Data Sheet" (Appendix G) will be completed in duplicate. One copy of the sample information form will accompany the composite samples to the drying rooms and TAES and one copy will be included in the project file at TAES/TSU.

Manure/compost Samples. Manure or manure compost samples will be double bagged in sealable plastic bags and marked with sample identification on the outside of both bags using a waterproof marker. The sample identification will identify the dairy and the manure/compost pile(s) from which the sample was taken. Plastic bags containing manure samples will be boxed and transported to TAES Laboratory, Stephenville, Texas. A "Compost Sample Data Sheet" (Appendix I) will be completed in duplicate. One copy of the sample information form will accompany the manure samples to the laboratory and one copy will be included in the project file at TAES.

Laboratory Analysis and Data Collection

A Test Group code is marked on the COC by the field staff to designate the type of analytes to be measured for each sample. Upon receipt of samples and COC, the laboratory staff member compares the time of collection and the shortest holding time for the required analyses against the time of receipt to ensure that sufficient time has been allowed to complete the analyses. When analyses are complete, the laboratory staff check again to see whether the samples were analyzed within the holding time. This can become an issue when quality control checks are not met and the analysis must be repeated. Laboratory staff consistently monitor the remaining time for analyses and work to ensure that samples are analyzed within holding time restraints.

Aliquots of each sample are used by the laboratory staff in running the various analytical procedures. The sample number is marked on all containers to which aliquots are transferred. Aliquots are filtered, as necessary, and analyzed as per standard operating procedures. Data pertaining to analyte measurements are recorded in bound personal logbooks, which are specific to each procedure and analyst. According to the type of analysis, measurement data produced in the laboratory is either printed out from the automated analytical equipment, read from screens on equipment and copied to Excel spreadsheets that calculate concentrations. Printouts of data from analytical equipment and from Excel spreadsheets are taped into the bound notebooks. Measurement data are copied from the notebooks to the computer database. Physicochemical data are downloaded from the databases and transferred electronically to the SAS database.

Backup/Disaster Recovery

The S drive and the network server are backed up daily to a tape drive at Stephenville. In the event of a catastrophic systems failure, the tapes can be used to restore the data. Data generated on the day of the failure may be lost, but can be reproduced from raw data in most cases.

Archives/Data Retention

Original data recorded on paper files and electronic format are stored for at least five years in climate controlled, fire-resistant storage area at the Stephenville Research Center.

Deficiencies, Non-conformances and Corrective Action Related to Chain-of-Custody

Deficiencies are defined as unauthorized deviations from procedures documented in the QAPP or other applicable documents. Non-conformances are deficiencies, which affect quality and render the data unacceptable or indeterminate. Deficiencies related to chain-of-custody include but are not limited to delays in transfer, resulting in holding time violations; incomplete documentation, including signatures; possible tampering of samples; broken or spilled samples, etc.

Deficiencies are documented in logbooks, field data sheets, etc. by field or laboratory staff and reported to the cognizant field or laboratory supervisor who will notify the TAES Project Manager. The TAES Project Manager will notify the TSSWCB QAO of potential non-conformances within 24 hours. The TAES Project Manager, in consultation with TSSWCB QAO (and other affected individuals/organizations), will determine if the deficiency constitutes a nonconformance. If it is determined the activity or item in question does not affect data quality and, therefore, is not a valid nonconformance, the CAR will be completed accordingly and closed. If it is determined a nonconformance does exist, the TAES Project Manager in consultation with the TSSWCD QAO will determine the disposition of the nonconforming activity or item and necessary corrective action(s); results will be documented on the CAR.

Corrective Action Reports (CARs) document: root cause(s); impact(s); specific corrective action(s) to address the deficiency; action(s) to prevent recurrence; individual(s) responsible for each action; the timetable for completion of each action; and the means by which completion of each corrective action will be documented. Notification of deficiencies documented by CARs will be included with quarterly progress reports. In addition, significant conditions (i.e., situations which, if uncorrected, could have a serious effect on safety or on the validity or integrity of data) will be reported to the TSSWCB immediately both verbally and in writing.

B4 ANALYTICAL METHODS

The analytical methods, associated matrices, and performing laboratories are listed in Table A7.1 of Section A7. Procedures for laboratory analysis will be in accordance with the most recently published edition of *Standard Methods for the Examination of Water and Wastewater*, the latest version of the *TCEQ Surface Water Quality Monitoring Procedures Manual*, 40 CFR 136, or other reliable procedures. Exceptions to this include analyses and sample matrices for which no regulated methods exist, or where EPA has not approved any method with adequate sensitivity. In this project, these methods include all of the analyses for soil, manure, compost, and forage tissue analytes because no methods have been approved by EPA. The analytical methods chosen to provide soils data include methods outlined in the Soil Science Society of America Soil Methods Book and those outlined in Attachment 3. The analytical methods chosen to provide forage tissue data and manure nutrient values are those outlined by the A.O.A.C. (1990. Official methods of analysis, 15th Ed. Association of Official Analytical Chemists, Wash. D.C.) and listed in Table A7.1.

Copies of TAES laboratory SOPs are retained by the TAES. Copies of TIAER SOPs are retained by TIAER.

Standards Traceability

All standards used in the field and laboratories are traceable to certified reference materials. Standards preparation is fully documented and maintained on-line (TIAER & TAES Stephenville) and in hard copies (TAES Stephenville) imbedded in the data sets for which they served as standards. Each documentation includes information concerning the standard identification, starting materials, including concentration, amount used and lot number; date prepared, expiration date and preparer's name. The reagent bottle is labeled in a way that will trace the reagent back to preparation.

Analytical Method Modification

Deficiencies, Non-conformance and Corrective Action Related to Analytical Methods

Deficiencies are defined as unauthorized deviations from procedures documented in the QAPP or other applicable documents. Non-conformance is deficiencies, which affect quality and render the data unacceptable or indeterminate. Deficiencies related to field and laboratory measurement systems include but are not limited to instrument malfunctions, blank contamination, quality control sample failures, etc.

Deficiencies are documented in logbooks, field data sheets, etc. by field or laboratory staff and are reported to the cognizant field or laboratory supervisor via a corrective action report (CAR). The supervisor notifies the TAES Project Manager if the deficiency has the potential of being a nonconformance. The TAES Project Manager will notify the TSSWCB QAO of the potential nonconformance within 24 hours.

The TAES Project Manager, in consultation with TSSWCB QAO (and other affected individuals/organizations), will determine if the deficiency constitutes a nonconformance. If it is determined the activity or item in question does not affect data quality and therefore, is not a valid nonconformance, the CAR will be completed accordingly and closed. If it is determined a nonconformance does exist, the TAES Project Manager in consultation with the TSSWCB QAO will determine the disposition of the nonconforming activity or item and necessary corrective action(s); results will be documented on the CAR.

CARs document root cause(s); impact(s); specific corrective action(s) to address the deficiency; action(s) to prevent recurrence; individual(s) responsible for each action; the timetable for completion of each action; and the means by which completion of each corrective action will be documented. Notification of deficiencies documented by CARs associated with non-conformances will be included with quarterly progress reports. In addition, significant conditions (i.e., situations which, if uncorrected, could have a serious effect on safety or on the validity or integrity of data) will be reported to TSSWCB both verbally and in writing.

B5 QUALITY CONTROL

Sampling Quality Control Requirements and Acceptability Criteria

Soils, compost and forage samples will be sampled in triplicate based on replicated field design. Wherever soil amendments (including compost) or vegetation treatments are applied, these will be done in triplicate in the field in order to ensure that extraneous error can be excluded through analyses of variance. If error exceeds 20%, these data will be excluded.

Field Split (water samples only) - A field split is a single sample subdivided by field staff immediately following collection and submitted to the laboratory as two separately identified samples according to procedures specified in the SWQM Procedures Manual. Split samples are preserved, handled, shipped, and analyzed identically and are used to assess variability in all of these processes. Field splits apply to conventional samples only and are collected on a 10% basis or one per batch, whichever is greater. The precision of field split results is calculated by relative percent difference (RPD) using the following equation:

$$RPD = \{ (X_1 - X_2) / \{(X_1+X_2)/2\} \} * 100$$

A 20% RPD criteria will be used to screen field split results as a possible indicator of excessive variability in the collection and analytical system. If it is determined that meaningful quantities of constituent (i.e., >AWRL) were measured and analytical variability can be eliminated as a factor, then variability in field split results will primarily be used as a trigger for discussion with field staff to ensure samples are being handled in the field correctly. Some sample results or batches of samples may be invalidated based on the examination of all extenuating information. Professional judgment during data validation will be relied upon to interpret the results and take appropriate action. The qualification (i.e., invalidation) of data will be documented on the Data Summary. Deficiencies will be addressed as specified in this section under Deficiencies, Nonconformance, and Correction Action related to Quality Control.

Laboratory Measurement Quality Control Requirements and Acceptability Criteria

Detailed laboratory QC requirements and corrective action procedures are contained within the TIAER laboratory quality assurance manuals (QAMs). The minimum requirements that all participants abide by are stated below. Lab QC sample results may be submitted with the laboratory data report (see Section A9.).

AWRL/Reporting Limit Verification

The laboratory's reporting limit for each limit will be at or below the AWRL. To demonstrate the ongoing ability to recover at the reporting limit, the laboratory will analyze a calibration standard (if applicable) at or below the reporting limit on each day EOF runoff samples are analyzed.

Two acceptance criteria will be met or corrective action will be implemented. First, calibrations including the standard at the reporting limit will meet the calibration requirements of the analytical method. Second, the instrument response (e.g., absorbance, peak area, etc.) for the standard at the reporting limit will be treated as a response for a sample by use of the calibration equation (e.g., regression curve, etc.) in calculating an apparent concentration of the standard. The calculated and reference concentrations for the standard will then be used to calculate percent recovery (%R) at the reporting limit using the equation:

$$\%R = CR/SA * 100$$

where CR is the calculated result and SA is reference concentration for the standard. Recoveries must be within 80-120% of the reference concentration.

When daily calibration is not required, or a method does not use a calibration curve to calculate results, the laboratory will analyze a check standard at the reporting limit on each day EOF runoff samples are analyzed. The check standard does not have to be taken through sample preparation, but must be recovered within 80-120% of the reference concentration for the standard. The percent recovery of the check standard is calculated using the following equation in which %R is percent recovery, SR is the sample result, and SA is the reference concentration for the check standard:

$$\%R = SR/SA * 100$$

If the calibration (when applicable) or the recovery of the calibration or control standard is not acceptable, corrective actions (e.g., re-calibration) will be taken to meet the specifications before proceeding with analyses of NPS samples.

The TIAER laboratory may report results of quantification checks with the data.

Laboratory Control Standard (LCS) - A LCS consists of analyte-free water spiked with the analyte of interest prepared from standardized reference material. The LCS is spiked into laboratory-pure water at a level less than or equal to the mid-point of the calibration curve for each analyte. The LCS is carried through the complete preparation and analytical process. The LCS is used to document the bias of the analytical process. LCSs are run at a rate of one per batch. Results of LCSs are calculated by percent recovery (%R), which is defined as 100 times the measured concentration, divided by the true concentration of the spiked sample.

The following formula is used to calculate percent recovery, where %R is percent recovery; SR is the measured result; SA is the true result

$$\%R = SR/SA * 100$$

Performance limits and control charts are used to determine the acceptability of LCS analyses. Project control limits are specified in Table A7.1.

Laboratory Duplicates - A laboratory duplicate is prepared in the laboratory by splitting aliquots of an LCS. Both samples are carried through the entire preparation and analytical process. LCS duplicates are used to assess precision and are performed at a rate of at least one per batch of 20 samples, or once per day, whichever is greater.

For most parameters, precision is calculated by the relative percent difference (RPD) of LCS duplicate results as defined by 100 times the difference (range) of each duplicate set, divided by the average value (mean) of the set. For duplicate results, X_1 and X_2, the RPD is calculated from the following equation:

$$RPD = \{ (X_1 - X_2)/\{(X_1+X_2)/2\} \} * 100$$

Performance limits and control charts are used to determine the acceptability of duplicate analyses. Project control limits are specified in Table A7.1.

Matrix spike (MS) - A matrix spike is an aliquot of sample spiked with a known concentration of the analyte of interest. Percent recovery of the known concentration of added analyte is used to assess accuracy of the analytical process. The spiking occurs prior to sample preparation and analysis. Spiked samples are routinely prepared and analyzed at a rate of 10% of samples processed, or one per batch whichever is greater. The MS is spiked at a level less than or equal to the midpoint of the calibration or analysis range for each analyte. Percent recovery (%R) is defined as 100 times the observed concentration, minus the sample concentration, divided by the true concentration of the spike.

The percent recovery of the matrix spike is calculated using the following equation in which %R is percent recovery, SSR is the observed spiked sample concentration, SR is the sample result, and SA is the reference concentration of the spike added:

$$\%R = (SSR - SR)/SA * 100$$

MS recoveries are plotted on control charts and used to control analytical performance. Measurement performance specifications for matrix spikes are not specified in this document, and MS data should be evaluated on a case-by-case basis.

Method blank - A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in the sample processing and analyzed with each batch. The method blank is carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination from the analytical process (see discussion of Method Blank under "Sample Containers" on page 24). The analysis of method blanks should yield values less than the reporting limit. For very high-level analyses, the blank value should be less then 5% of the lowest value of the batch, or corrective action will be implemented.

Additional method specific QC requirements - Additional QC samples are run (e.g., special LCS studies, continuing calibration samples) as specified in the methods. The requirements for these samples, their acceptance criteria, and corrective action are method-specific.

Deficiencies, Non-conformances and Corrective Action Related to Quality Control

Deficiencies are defined as unauthorized deviations from procedures documented in the QAPP. Non-conformances are deficiencies, which affect quality and render the data unacceptable or indeterminate. Deficiencies related to quality control include but are not limited to field and laboratory quality control sample failures.

Deficiencies are documented in logbooks, field data sheets, etc. by field or laboratory staff and are reported to the cognizant field or laboratory supervisor via a corrective action report (CAR). The supervisor notifies the TAES Project Manager if the deficiency has the potential of being a nonconformance. The TAES Project Manager will notify the TSSWCB QAO of the potential nonconformance within 24 hours.

The TAES Project Manager, in consultation with TSSWCB QAO (and other affected individuals/organizations), will determine if the deficiency constitutes a nonconformance. If it is determined the activity or item in question does not affect data quality and therefore, is not a valid nonconformance, the CAR will be completed accordingly and closed. If it is determined a nonconformance does exist, the TAES Project Manager in consultation with the TSSWCB QAO will determine the disposition of the nonconforming activity or item and necessary corrective action(s); results will be documented on the CAR.

CARs document root cause(s); impact(s); specific corrective action(s) to address the deficiency; action(s) to prevent recurrence; individual(s) responsible for each action; the timetable for completion of each action; and the means by which completion of each corrective action will be documented. Notification of deficiencies documented by CARs associated with non-conformances will be included with quarterly progress reports. In addition, significant conditions (i.e., situations which, if uncorrected, could have a serious effect on safety or on the validity or integrity of data) will be reported to the TSSWCB both verbally and in writing.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION AND MAINTENANCE

EOF sampling equipment (manufactured by TCE Ag Engineering, College Station, and consist of 1 m X 1 m exclusions, funnel, hose and buried barrel that can be removed, sampled and liquid measured) is inspected and tested upon receipt and is assured appropriate for use by the project engineer. Equipment records are kept on all field equipment and a supply of critical spare parts is maintained. If problems cannot be remediated on-site by the project engineer during the inspection, required adjustments or repairs will be made as soon as possible by the project engineer at the College Station TCE Ag Engineering facilities.

All laboratory tools, gauges, instrument, and equipment testing and maintenance requirements are contained within laboratory standard operating procedures. Testing and maintenance records are maintained and are available for inspection. Instruments requiring daily or in-use testing include, but are not limited to, water baths, ovens, autoclaves, incubators, refrigerators, and laboratory-pure water. Critical spare parts for essential equipment are maintained to prevent downtime. Maintenance records are available for inspection.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Detailed laboratory calibrations are contained within the standard operating procedures. TAES, TIAER and TSU standard operating procedures identify all tools, gauges, instruments, and other sampling, measuring, and test equipment used for data collection activities affecting quality that must be controlled and, at specified periods, calibrated to maintain bias within specified limits. Calibration records are maintained, are traceable to the instrument, and are available for inspection by the TSSWCB. Equipment requiring periodic calibrations includes, but are not limited to, thermometers, ICP, C-N analyzers, HPLC, ovens, pH meters, balances, incubators, analytical instruments. Deficiencies will be resolved by the technicians responsible for the equipment or by company engineers and evidence of these calibrations will be maintained by the technicians and will be available for inspection at any time.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

All new batches of field and laboratory supplies are inspected and tested before use to ensure that they are adequate and not contaminated. Supplies are inspected upon receipt to confirm shipping condition, quality requirements, and quantity. Chemicals, reagents and standards are logged into an inventory database that documents grade, lot number, manufacturer, dates received, opened & emptied. All reagents shall meet ACS grade or equivalent where required. Acceptance criteria are detailed in organization's standard operating procedures. The laboratory standard operating procedures provide additional details on acceptance requirements for laboratory supplies and consumables.

B9 NON-DIRECT MEASUREMENTS

Literature files collected by the project manager and other members of the project team will be consulted for non-direct data sources. Additional sources will be accessed on Agricola via the Texas A&M libraries and its electronic facilities at College Station. These data will be used to compare results and identify parallels to the project, an accepted form of self-evaluation fostered by the American Society of Agronomy and its journals. Resources will include desktop computers utilized to access the A&M libraries via the internet as well as file cabinets and shelves in each individual's office. Using these resources, the project manager will assist with project members in determining validity and operating conditions proposed in the literature and measured in the field.

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B10 DATA MANAGEMENT

Data Management Process

Section B3 contains a detailed discussion of how samples are handled from collection through delivery to the laboratories. Included within that discussion is a description of how station information is taken and recorded on COC and other data forms. This section continues with the manner in which data are handled by TAES until they are submitted to TSSWCB. In addition, this section outlines the data management associated with samples submitted to the TAES Stephenville Laboratory.

Personnel

TCE personnel responsible for data generation and collection are listed by position below.

Edge-of Field Water Runoff Monitoring Staff are responsible for correctly recording forage and buffer strip EOF runoff and ensuring the data are sent to the data manager.

TAES TSU, TCE & TIAER personnel are responsible for data generation and management listed by position below.
Laboratory Analysts are responsible for collection of analytical results from automated analyzers and analytical procedures, correctly transferring those data to electronic logbooks and then to store data sheets in hardcopy form.

Personnel responsible for data validation, input, and transfer are listed below.

Field Supervisors, Laboratory Manager and QAO- responsible for screening data for anomalies and mistakes before and after entry into the TAES databases

Laboratory Staff and Data Entry Technicians - responsible for entry of COC, field, and laboratory data into a Microsoft Excel entry table

Laboratory Managers - responsible for verifying that all identification (COC) and laboratory data entered for each sample are correct according to the information on the COC, field data sheets, and personal laboratory notebooks, and transferring the data from the Access entry table to the Access water quality database. Also responsible for making corrections and changes to the database and maintaining an audit trail of the changes.

Project Manager s (TSU & TAES)- responsible for importing data from the Excel database into the TAES database and subsequent statistical analyses (SAS or MSTATC).

Data Management Plan Implementation – The data will pass from the laboratory manager, to the respective TAES/TSU project leaders who will be responsible for data analysis, interpretation, write-up and subsequent storage.

TAES/TSU/TCE Water, soil & plant Quality Data Entry - As described in Section B3, generated data entered on the COCs and in laboratory logbooks are passed on to project managers. Afterwards, a data analyst reviews the COCs for correctness, abnormalities, and problems. Dairy names (or numbers), plot descriptions, appropriateness of data values, legibility of writing, completeness of data, dates and times, bottle numbers, start and end times of composited samples, comments and all other data on the sheets are reviewed. Any questions or abnormalities are investigated, relying largely on field and laboratory data sheets, general maintenance sheets, field technicians, laboratory notebooks, sampler printouts, compositing program printouts, and laboratory personnel. Any errors are crossed out with a single line, initialed and dated and the correct data are added. Corrective action reports are completed, as appropriate.

After receiving a batch of COCs, the Laboratory Managers open the electronic (Excel) entry table and verifies that all data entered for each sample are correct according to the information on the COC form and data entry sheets. After checking all the data on the forms, the Laboratory Managers initial the forms and sends them to the Project Managers, who file them in a three-ring binder sequentially by sample number. COC binders are maintained on file for at least five years.

After a batch of COCs has been verified, the Laboratory Managers transfer the data to Excel entry table and notifies the Data Manager that new data have been added. Before data are analyzed for projects, they are screened for outliers and other abnormalities by a data analyst. All necessary records are checked to resolve problems. If errors are found, the data are corrected on the COC or data entry form with a single line and the initials and date. The Laboratory Manager and Project Manager are consulted before and after correction.

TAES/TCE/TSU/TIAER Data Errors and Loss

Migration/Transfer/Conversion - File transfer protocols used for ensuring proper exportation of data from the TAES database include the data quality assurance procedures integral to the data system.

Backup/Disaster Recovery - The Unix drive and the network server are backed up daily to a tape drive located in a climate controlled, fire-resistant storage area on the Tarleton State University, Texas A&M and TAES Stephenville campuses. In the event of a catastrophic systems failure, the tapes can be used to restore the data in less than one day's time. Data generated on the day of the failure may be lost, but can be reproduced from raw data in most cases.

Archives/Data Retention - Original data recorded on paper files are stored for at least five years. Data in electronic format are stored on tape drives. Complete electronic data sets are archived on tape backup and retained on the Tarleton State University campus in a fire-resistant storage area managed by the Tarleton CIS department.

TAES/TCE/TSU/TIAER Record Keeping and Data Storage

Individual laboratory notebooks, which contain printouts of laboratory data and hand written observations and data, are kept by individual analysts at TIAER. When lab notebooks are filled, they are stored for at least five years by the laboratory manager in hardcopy form. TSU/TCE/TAES laboratories keep their electronic data on personal computers for the duration of the project and then in hardcopy files for 5 years after the project. The original field data sheet is filed in a three-ring binder, according to site location and project, and stored in the field operations room for at least five years. COCs and attached documents are stored in numerical order in three-ring binders in the TAES Data Manager's office for at least five years. All electronic records are stored for a minimum of five years on personal computers and TAES/TSU/TCE/TIAER fire-proof cabinets.

TAES/TCE/TSU/TIAER Data Handling, Hardware, and Software Requirements

The TAES/TSU/TIAER laboratories employ the following data handling software on personal computer stations for data on many of the analyzed parameters:

- Total P will be analyzed using a Spectro ICP-AES at Tarleton: The SPECTRO Smart Analyzer Vision software is the flexible and powerful user interface to the SPECTRO CIROS CCD ICP spectrometers. It combines simple "one-click" routine operation with new functionalities to take full advantage of the unique SPECTRO CIROSCCD analytical capabilities. The intuitive MS-Office-like design and interactive help wizards make the SPECTRO Smart Analyzer Vision easy to use and self-explanatory. Complete instrument control, whether for manual or unattended automatic operation, along with logging of commands and events, results in increased productivity and reliable operation. Operates under Windows XP Professional and is US FDA 21 CFR Part 11 compliant.

- Total C & N will be analyzed using an Elementar Macro analyzer: Windows software operating under Windows XP Professional operates and evaluates the analyzer. Integrated maintenance software with automatic leak tests, wake/sleep functions, LIMS connectivity and direct data export to Excel are present. An optional module to make the unit US FDA CFR Part 11 compliant is available, but we plan to keep all data in disk and hard copy format for 5 years.

The TAES/TSU/TIAER Laboratory Managers are responsible for review of calculations and charts made by these programs. Biometric analyses are computed using Excel spreadsheets and SAS or MSTATC programs. Microsoft Excel is used for general spreadsheet computation and laboratory control charting of quality control parameters.

The TAES/TSU/TCE field operations staff uses data sheets to record field & drying room measurements (see Appendix G).

The TIAER laboratory data are stored in an Excel databases, then are uploaded to a SAS database on a Unix server. SAS programs are used to screen data for outliers, compile data sets, and analyze data trends. TAES and TSU laboratory data will be stored in Excel files (hardcopies for 5 years, on personal computers and the TAES SHARE drive for the duration of the project).

As part of the data review process, checks on written data compared to TAES data in the SAS database are used to ensure that the hardware/software configurations are correctly storing and retrieving data.

Information Resource Management Requirements

Data submitted to TSSWCB will be screened by the TAES Quality Assurance Officer prior to submission to ensure that all data records use the proper format and contain all required information. A data review checklist will be utilized (see Appendix D).

Information Dissemination - Submission of the data produced for this project will be transferred from TAES/TCE/TSU to TSSWCB as deemed appropriate.

C1 ASSESSMENTS AND RESPONSE ACTIONS

The following table presents the types of assessments and response actions for data collection activities applicable to the QAPP.

Table C1.1 Assessments and Response Requirements

Assessment Activity	Approximate Schedule	Responsible Party	Scope	Response Requirements
Status edge-of-field Oversight, etc.	Continuous	TCE project engineer	Monitoring of the project status and records to ensure requirements are being fulfilled	Report to TSSWCB in Quarterly Report
Edge-of-field Systems Audit	Biennially	TAES & TSSWCB QAO	Field sampling, handling and measurement; facility review; and data management as they relate to NPS	30 days to respond in writing to the TAES to address corrective actions
Laboratory Inspection	Biennially	TAES QAO & TSSWCB QAO	Requirements appearing in lab SOPs and QAPP, ISO/IEC Guide 25, applicable EPA methods and Standard Methods, 40 CFR 136, and other documents applicable to NPS programs including portions of the Texas Administrative Code and the Code of Federal Regulations.	30 days to respond in writing to the TAES to address corrective actions

Corrective Action

The TAES Project Manager is responsible for implementing and tracking corrective action procedures as a result of audit findings. Records of audit findings and corrective actions are maintained by the TAES project manager. Corrective action documentation will be submitted to the TSSWCB on a quarterly basis with the Progress Report.

If audit findings and corrective actions cannot be resolved, then the authority and responsibility for terminating work are specified in agreements in contracts between participating organizations.

Corrective actions include identification of root causes and a methodology for correcting the problems. The effect of the problem on the quality of the data is ascertained and documented on the CAR. The programmatic impact (up to and including the removal of data from the database) of the deficiency must be ascertained and documented. The impact of deficiencies must be made on a case-by-case basis in consultation with the TAES Project Manager and TSSWCB QAO.

C2 REPORTS TO MANAGEMENT

Laboratory Data Reports

Laboratory data reports contain the results of all specified QC measures listed in section B5, including but not limited to laboratory duplicates, laboratory control standards, and calibrations. This information is reviewed by the TAES QAO and compared to the pre-specified acceptance criteria to determine acceptability of data before submitting it to analyses and storage. This information is also available for inspection by TSSWCB.

Reports to TAES Project Management

TAES/TCE/TSU project participants submit written quarterly progress reports to the TAES Project Manager concerning the status of each project task, including data collection activities, for which they are responsible. Any issues or problems associated with the quality of the data are reported to the TAES Project Manager through the use of Corrective Action Reports.

Reports to TSSWCB Project Management

Quarterly Progress Report – TAES will summarize the TAES/TCE/TSU activities for each task; reports problems, delays, and corrective actions; and outlines the status of each task's deliverables.

Monitoring Systems Review Audit Report/Laboratory Audit Report and Response - Following any audit performed by TSSWCB, a report of findings, recommendations and response is sent to the TSSWCB project manager in the quarterly progress report.

Final Project Report - Summarizes the TAES's and subcontractor activities for the entire project period including a description and documentation of major project activities; evaluation of the project results and environmental benefits; and a conclusion.

D1 DATA REVIEW, VERIFICATION, AND VALIDATION

All field and laboratory data will be reviewed and verified for integrity and continuity, reasonableness, and conformance to project requirements, and then validated against the data quality objectives, which are listed in Section A7. Only those data, which are supported by appropriate quality control data and meet the data quality objectives defined for this project will be considered acceptable, and will be reported to TAES and TSSWCB.

The procedures for verification and validation of data are described in Section D2, below. The TAES/TSU/TCE Field Supervisors are responsible for ensuring that field data are properly reviewed and verified for integrity. The TAES/TSU/TIAER Laboratory Managers are responsible for ensuring that analytical laboratory data are scientifically valid, defensible, of acceptable precision and accuracy, and reviewed for integrity. The TAES Project Manager will be responsible for ensuring that all data are properly reviewed and verified, and submitted in the required format to the project database. The TAES Project Manger/QAO is responsible for validating the data. Finally, the TAES Project Manager/QAO, is responsible for validating that all data to be reported meet the objectives of the project and are suitable for reporting to TSSWCB.

D2 VERIFICATION AND VALIDATION METHODS

All field and laboratory data will be reviewed, verified and validated to ensure they conform to project specifications and meet the conditions of end use as described in Section A7 of this document.

Data review, verification, and validation will be performed using self-assessments and peer and management review as appropriate to the project task. The information to be reviewed, verified, and validated (listed by task and responsible party in Table D2.1) is evaluated against technical and project specifications and checked for errors, especially errors in calculations, data reduction, and transcription. Potential errors are identified by examination of documentation and by manual (and computer-assisted) examination of corollary or unreasonable data. If a question arises or an error is identified, the manager of the task responsible for generating the data is contacted to resolve the issue. Issues, which can be corrected are corrected and documented. If an issue cannot be corrected, the Project Engineer or TSU Project Coordinator consults with TAES Project Manager to establish the appropriate course of action, or the data associated with the issue are rejected. Field and laboratory reviews, verifications, and validations will be documented.

Data validation tasks to be addressed by TAES/TCE/TSU include, but are not limited to, the confirmation of lab and field data review, evaluation of field QC results, additional evaluation of anomalies and outliers, analysis of sampling and analytical gaps, and confirmation that all parameters and sampling sites are included in the QAPP. Any suspected errors or anomalous data must be addressed by the manager of the task associated with the data before data validation can be completed. Any issues requiring corrective action must be addressed, and the potential impact of these issues on previously collected data will be assessed. Finally, the TAES Project Manager validates that the data meet the data quality objectives of the project and are suitable for reporting to TSSWCB. Pertinent information having to do with inconsistencies with reporting limit specifications; failures in sampling methods and/or laboratory procedures resulting in unavailable data, etc. will be provided on the Data Summary when the data are submitted to TSSWCB.

Table D2.1 Data Review, Verification, and Validation Tasks

Task	Verification	Validation	Responsibility
Field data reviewed for conformance with data collection, sample handling and chain of custody, analytical and QC requirements	Y		Field Operation Supervisor
Post-calibrations checked to ensure compliance with error limits	Y		Field Operation Supervisor
Field data calculated, reduced, and transcribed correctly	Y		Field Operation Supervisor
Laboratory data reviewed for conformance with data collection, sample handling and chain of custody, and analytical and QC requirements to include documentation, holding times, sample receipt, sample preparation, sample analysis, project and program QC results, and reporting	Y		Laboratory Managers
Laboratory data calculated, reduced, and transcribed correctly	Y		Laboratory Managers
Reporting limits consistent with requirements for Ambient Water Reporting Limits.	Y	Y	TIAER Laboratory Manager
Analytical data documentation evaluated for consistency and/or improper practices	Y	Y	Laboratory Managers
Analytical QC information evaluated to determine impact on individual analyses	Y	Y	Laboratory Managers
All laboratory samples analyzed for all parameters	Y	Y	Laboratory Managers
Data set (to include field and laboratory data) evaluated for reasonableness and if corollary data agree	Y	Y	Laboratory Managers, TAES QAO
Data review, verification, and validation performed and deviations documented		Y	Laboratory Managers, TAES QAO
Outliers confirmed and documented		Y	TCE Project Engineer, TSU Project Coordinator & TAES QAO
Field QC acceptable (e.g., field splits)		Y	Laboratory Managers, TCE Project Engineer, TSU Project Coordinator & TAES QAO
Sampling and analytical data gaps checked and documented		Y	TCE Project Engineer, TSU Project Coordinator & TAES QAO
Verification and validation confirmed. Data meets conditions of end use and are reportable		Y	TAES Project Manager

D3 RECONCILIATION WITH USER REQUIREMENTS

The data collected in this project can be used as part of efforts to address nonpoint source pollution issues in impaired watersheds. Data that do not meet requirements will not be submitted to TAES or TSSWCB nor will be considered appropriate for any of the uses noted above.

Samples collected from this project will be analyzed by the TAES, TSU, and TIAER laboratories and reported to TSSWCB for evaluation of the measured reductions of phosphorus in soils and EOF runoff. The percentage of phosphorus removal or sequestration achieved, as a result of the vegetation system performance or soil type, will be one of several criteria examined by TAES/TSU/TCE in the design and sizing of soil phytoremediation systems to be utilized on other dairies.

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APPENDIX A. Work Plan

PHYTOREMEDIATION of EXCESSIVELY HIGH PHOSPHORUS SOILS and SUBSEQUENT REDUCED PHOSPHORUS RUNOFF from CROPLANDS RECEIVING DAIRY WASTE on the NORTH BOSQUE RIVER
Texas State Soil and Water Conservation Board
FY03 CWA Section 319(h)
WORK PLAN

Problem Need/Statement

General Project Description

We propose to develop and demonstrate remedial best management practices (BMP) for both abandoned currently used waste application fields that will bring soil P levels back to safe levels. These BMPs may also allow dairymen to harvest P in amounts equal to that contained in dairy waste applied to fields without risking soil-P buildup. In either case, there is an urgent need to intercept P-rich runoff from these fields by establishing permanent vegetation buffer strips between tilled soils and streams.

Year-round forage systems that extract plant available P from soils can reduce, recycle and stabilize excess P in Windthorst soils (the predominant sandy loam used for growing forages in the watershed). Developing and promulgating these crop systems involves maximizing land-occupation time (compatibility of winter and summer crops) and finding species whose high yields and herbage-P concentrations combine to export the greatest possible amount of soluble soil-P.

The goal is to develop and demonstrate year-round forage systems that can reduce P loads on cropping land that soon will or already exceeds safe levels of plant-available P on the North Bosque River drainage. Until soil-P levels are brought down to acceptable levels, vegetative buffer strips will intercept most run-off P. These solutions will be cost-effective and acceptable to TSSWCB under permit and TMDL programs. Widespread implementation of these forage systems will promote sustainability of the dairy industry and improve environmental quality of surface water.

Tasks, Objectives, Schedules, and Estimated Costs

Task 1: Document/demonstrate a measurable rate of P removal via forages from crop fields with histories of excessive dairy waste application.

Costs: Federal $177,859; Match $95,182 ; Total $273,041

Objective: To reduce surface water contamination in the North Bosque River from soil-applied P of dairy manure origin.

Subtask 1.1: Locate three crop fields on the upper North Bosque River that have an excess of 200 ppm plant-available P due to historical applications of dairy wastes. Measure all components (both stable and plant-available) of P in the soil.

Subtask 1.2: On these fields, determine optimal season length and P-extraction of cool season winter grasses and legumes on small plots by measuring both forage yields and P concentrations. Individual species will be tested years 1-2.

Subtask 1.3: On these same fields but distinct plots, determine optimal season length and P-extraction of warm season winter grasses and legumes on small plots by measuring both forage yields and P concentrations. Individual species will be tested years 1-2.

Subtask 1.4: Based on results from subtasks 1.2 and 1.3, design and demonstrate year-round forage production systems that have compatible growing seasons (do not overlap) and extract the maximum amount of plant-available P from the these crop fields. Determine which systems extract the greatest amount of soil-P and determine if the plant-available P extraction affects stable P in the soils. These systems will be demonstrated in years 2 & 3.

Subtask 1.5: Prepare quarterly and final reports. The final Report will be submitted to the TSSWCB, via CD, at the culmination of the project. The report will include information from the FY02-5 project. The TSSWCB project manager will set dates for the reports. (Month 1 to Month 24)

Subtask 1.6: The technician will attend quarterly meeting with the TSSWCB project manager to review project status, deliverables, etc. (Month 1 to Month 24)

Deliverables

- QAPP and Reports of individual forage or grain crop P concentration.
- Reports of forage systems P extraction (a function of forage P concentration and herbage yield) and the effects on soil EP.
- Quarterly and final reports documenting project status.
- Semi-annual reports will be developed by the TSSWCB project manager to be submitted to EPA.

Task 2: Develop and test easily established vegetative buffer strips that harvest or stabilize soil-P in surface water runoff moving from dairy waste application fields to streams, rivers or other drainages.

Costs: Federal <u>$40,000</u>; Match <u>$21,000</u>; Total <u>$61,000</u>

Subtask 2.1: Design and test first tier buffers composed of harvestable (hay) material with high soil-P extraction (yield X P concentration) potential that will catch and recycle surface runoff from croplands high in water-soluble P of dairy waste origin.

Design and establish demonstrations (at least one site per soil type) will take place year 1; years 2-3 will involve soil, forage, and edge-of-field monitoring.

Subtask 2.2: Design and demonstrate second tier buffer strips for the specific soils, drainages and climate that will foster year-round buffering utilizing mostly (but not exclusively) native vegetation. These will consist of secondary tiers of mostly perennial species with ecologically stable environments. Designing and establishing demonstrations (at least one site per soil type) will take place year 1; years 2-3 will involve soil, forage, and edge-of-field monitoring.

Deliverables:

- Buffer designs, and producer-oriented publications with buffer-establishment and management instructions.

Task 3: Based on results from the previous tasks, model and demonstrate forage systems, first tier and second tier buffers to accurately predict maximum soil-P removal and stabilization rates (and, consequently, maximum tolerable additional dairy waste-P) for the soils in the North Bosque.

Costs: Federal $0; Match $56,000; Total $56,000

Subtask 3.1: Develop, field test, and demonstrate to dairy producers and their neighbors currently accepting dairy wastes on their fields a crop field-to-stream model of how to maximize removal and stabilization of dairy waste P. This will entail utilizing results collected years 1 & 2 and will therefore be developed and field tested year 3.

Deliverables:

- Theoretical models based on concrete experiences and, finally, producer-oriented publications that propose BMP's for bioremediation of high P soils.

Task 4: Model the economics associated with implementation of the integrated approaches outlined above. Data collection for this will take place from year 1-3 but final modeling will occur only during year 3.

Costs: Federal $21,000; Match $000; Total $21,000

Deliverables:

- Producer-oriented documents for scenarios from Tasks 1-3 outlining costs and benefits of the BMP's being promulgated.

Coordination, Roles and Responsibilities:

Participating Agencies and Organizations along with their roles in this project include:

- **Texas Agricultural Experiment Station**. Dr. **Jim Muir**, forage and range ecologist, will coordinate the project as well as provide support for evaluating forage and buffer strip systems for remediating high-P soils, safely applying dairy manure/sludge/compost to cropland, and restricting surface runoff of P from animal wastes. The soil and forage laboratory at the Stephenville can test for phosphorus and other nutrients.
- **Tarleton State University**. Dr. **David Weindorf**, soil scientist, will be in charge of the soils aspects of tasks 1 & 2. His soils lab is also available for any analyses needed in monitored or tested fields. Dr. **Roger Wittie**, range scientist, will contribute his knowledge of buffer strips and native vegetation.
- **Texas Cooperative Extension.** The agronomist for District 8 will provide invaluable contacts in the field as well as his/her crop production knowledge in designing year-round forage systems that optimize P extraction and recycling.
- **Texas Cooperative Extension.** Dr. **Saqib Mukhtar** will bring his edge of field monitoring expertise to measure the deliverables resulting from management changes.

Measures of Success:

- We will quantity of manure-P that can be potentially recycled
- We will measure the decrease in both total and water-soluble soil P on demonstration fields with high P from dairy manure.
- Edge-of-field P runoff will be reduced on production crop fields that implement the developed and demonstrated BMPs.

APPENDIX B. Sampling Process Design and Monitoring Schedule (Plan)

Sample Design Rationale

The sample design is scheduled to provide data to characterize changes in soil phosphorus concentrations in fields receiving/having received dairy waste. Changes in phosphorus and other nutrients in the edge-of-field and various matrices associated with dairy waste application will be monitored before and after the implementation of phosphorus reduction vegetative strategies or soil types.

Site Selection Criteria

This project involves the collection of non-ambient data that will be used to gauge the effectiveness of the vegetative strategies or soil types on surface water runoff quality. The data collection effort involves monitoring nutrient content of samples associated with various aspects of dairy manure, soils and vegetation rather than sites that are representative of ambient water quality conditions.

Wet Weather Sampling at Edge-of-field and Intermittent Channel Stations

At the downstream end of each plot (replicated treatment plots set-up by the TSU Range Scientist and Project Manager), a 1x1m sub-plot will be established. Each sub-plot will be isolated from overland flow, using 15-cm metal borders, installed 10-cm above and 5-cm below the ground level. At the down slope end of each sub-plot, a V-shaped metal gutter will be used to capture all the natural rainfall runoff into a 80-L capacity plastic tub. The tub capacity is sufficient to hold up to ~7.5-cm runoff [estimated for the hydrologic soil conditions and land use management using the SCS Curve Number (USDA-NRCS, 1972)] resulting from a 25-yr, 24-h rainfall amount of ~18.3-cm (National Weather Service, 1961) for the study area. A trench will be dug and two rectangular tubs, one inserted into the other (fitting tightly), will be buried up to ¾ of their height near the V-shaped gutter outlet. Any cavities between the hole and the tubs will be filled with soil and the inner tub will be covered with a lid. A10-cm, diameter removable PVC pipe (connected with couplers) will be used to convey runoff from the gutter to the inner tub through a hole drilled in the side wall of both tubs. A tipping bucket rain gage with a data logger (Onset Computer Corp., Pocasset, MA) will be installed at each site to record natural rainfall amounts.

After every runoff producing natural rain event, the PVC pipe will be disconnected, the inner tub will be removed and its contents (water and sediment) will be weighed to determine runoff mass and volume. Runoff pH and electrical conductivity will be determined in the field using a pH and conductivity probe. From each tub, a thoroughly mixed 250-mL composite sample will be collected in plastic bottles. Laboratory analyses will be conducted to determine concentrations of TKN, TP, DP, TS and TSS from each sample.

Soil, Forage and Manure Samples.

Soil samples in trial and demonstration areas will be collected on an annual basis and will be analyzed using the routine analysis by the TAES Research Laboratory in Stephenville, TX. Soil samples used for mapping P in dairy soils will be collected only once at the beginning of the project. Analytes to be analyzed include TP, extractable K, TN, pH and TC.

Forage samples will be collected just prior to harvest of each crop. Samples will be collected using a 4 ft^2 (0.37 m^2) sampling frame randomly placed at least three different locations within each land forage and buffer plot. Vegetation will be cut to a height consistent with the harvest equipment. Forage yield will be estimated by TAES staff on a dry matter basis by weighing all sample material from each frame. Three sub-samples from each sample will be weighed and put in a drying oven at 131° F (55° C) until weight change ceases. Sub-samples will then be re-weighed to obtain a dry weight. The dry weight of each sub-sample will be divided by its fresh weight to obtain an estimate of original percent moisture. After drying and re-weighing, a composite sample of the three subsamples will be collected. For tissue analysis, the composite sample from each forage/buffer plot will be sent to the TAES Stephenville Laboratory for analysis of TP and TP. Tissue analysis along with yield values will be used to estimate the removal of N and P as forage harvested from each plot.

Manure/compost samples will be collected prior to application to fields if and when this occurs during routine dairy operations. In each sampling event, five or more sub-samples will be collected and composited into one sample for analysis. The samples will be analyzed by the TAES Stephenville Laboratory for TP and TN. The results of the analysis will aid in making appropriate recommendations regarding dairy manure applications on application fields and in compliance with CNMP provisions.

APPENDIX C. Chain-of-Custody Forms

TiAER

CHAIN OF COUSTODY

Page __ of __

Project Manager/Person(s) Requesting Sample	Sampler(s)

Project Code	Sample No.	Test Group Code	Date	Time	Site ID	Sample Type	Preservative	Containers	Comments (if applicable)	(if applicable)	(if applicable)	Disposed	T= 0-6C	pH checked
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			
									bottles:	end time/date:	Preservative deviation:			

Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:

Sample Types: G=Grab SG=Storm Grab P= Periphytometer/growth
O= Other F=Flow Based S=Sequential M= Multisonde BS= Bacteria storm

Container types: P= Plastic, D=Dark F=Filter
M=Multiple, G=Glass V=VOA, S=sterile

revised 12/16/04 mm

	initials	date
Data entry:		
Field review:		
Lab review:		

Preservatives: A=acid <pH 2; Y=syringe/field filtered; I=Ice or frozen; O= other

Texas Institute for Applied Environmental Research
Box T-0410, Stephenville, TX 76402, Tarleton State University 254-968-9570, 254-968-9560

APPENDIX D. Data Review Checklist

	Y, N, or N/A
Data Format and Structure	
A. Is the file in the correct format (e.g. ASCII pipe delimited)?	____
B. Are there any duplicate Tag Id numbers?	____
C. Are the Tag prefixes correct?	____
D. Are all Tag Id numbers 7 characters?	____
E. Are sampling Dates in the correct format, MM/DD/YYYY?	____
F. Is the sampling Time based on the 24 hour clock (e.g. 13:04)?	____
G. Is the Comment field filled in where appropriate (e.g. unusual occurrence, sampling problems, unrepresentative of ambient water quality)?	____
H. Source Code 1, 2 and Program Code used correctly and are valid?	____
I. Is sampling date in Results file the same as those in the Events file?	____
J. Values represented by a valid parameter (STORET) code with the correct units and leading zeros?	____
K. Are there any duplicate STORETs for the same Tag Id?	____
L. Are any invalid symbols in Greater Than/Less Than (GT/LT) field	____
M. Are any tag numbers in the Results file that are not in Events file?	____
N. Are confirmed outliers identified with a "1" in the remarks field?	____
Data Quality Review	
A. Are all the values reported at or below the appropriate AWRL?	____
B. Have the outliers been verified?	____
C. Checks on correctness of analysis or data reasonableness performed? e.g.:Is ortho-phosphorus less than total phosphorus? Are dissolved metal concentrations less than or equal to total metals?	____
D. Have at least 10% of the data been reviewed against the field and laboratory data sheets?	____
E. Are all STORET codes in the data set listed in the QAPP?	____
F. Are all stations in the data set listed in the QAPP?	____

Documentation Review

A. Are blank results acceptable as specified in the QAPP? ____________

B. Were control charts used to determine acceptability of field duplicates? ____________

D. Were there any failures in sampling methods and/or deviations from sample design requirements that resulted in unreportable data? If yes, explain on next page. ____________

E. Were any failures in field or laboratory measurement systems not resolvable and resulted in unreportable data? If yes, explain on next page. ____________

Describe any data reporting inconsistencies with AWRL specifications. Explain failures in sampling methods and field and laboratory measurement systems that resulted in data that could not be reported to the TSSWCB. (attach another page if necessary):

Date Submitted to TSSWCB:
TAG Series:
Date Range:
Data Source:
Comments:

TAES's Data Manager Signature:

Date:

Appendix E

Corrective Action Report

CAR #: ______________________________

Report Initiation Date: ____________________ **Area/Site:** ____________________________

Reported by: ____________________________ **Analyte/Activity:** ____________

State the nature of the problem, nonconformance or out-of-control situation: ____

_

____

Affected sample #s / date(s) of sample collection[1]: ______________________________________

Project(s): ________________________________ Attached documentation: NA COC FDS SampLink Flow

Possible Causes and Corrective Actions Taken / Recommended:

____

____

CAR routed to: ________________________________ Date: __________________________

____

Supervisor: Circle one: **Tier 1** (does not affect final data integrity) **Tier 2** (possibly affects final data integrity)

Corrective Actions (If actions are to be taken, include Responsible Party[2] and proposed completion date, where appropriate)

For specific incident: Taken To be taken ____

____

To prevent recurrences: Taken To be taken ____

____

Effect on data quality: ____

Responsible Supervisor: ______________________________________ Date: ________________

Concurrence: Program/Project Manager: ______________________________ Date: ________________

(Tier 2 CARs only) Quality Assurance Officer: ________________________ Date: ____________

[1] For storm samples, use date of the beginning bottle of affected sample. If appropriate, state whether a storm grab was collected.

[2] Party responsible for implementing corrective action is also responsible for notifying QAO of completion and outcome of corrective action

Q-105-1, rev. 1

Appendix F
Field Data Reporting Form

Station ID **Date** **Time**

Sample ID **COC Number**

Collector Name(s)

Collecting Agency

Days since last significant rainfall:

Weather Observations:

Other Observations:

Appendix G

Forage Sample Data Sheet

Date ____________________

Location ____________________ **Harvest method** ____________________

Collector ____________________ **Area harvested** ____________________

Experiment ____________________

Plot #	Rep	Species	Green Plot Wt	Green Sub-sample	Dry Sub-sample	DM %	DM Plot

Appendix H

Soil Sample Data Sheet

Date ______________________

Location ______________________ ______________________________

Collector ______________________ ______________________________

Experiment ______________________

Dairy	ID #	Depth	Composite Status				

Appendix I

Compost Sample Data Sheet

Date ____________________

Location ____________________ ______________________________

Collector ____________________ ______________________________

Experiment ____________________

Dairy	ID#	Depth	Composite Status				

ATTACHMENT 1 Example Letter to Document Adherence to the QAPP

TO: (name)
(organization)

FROM: (name)
(organization)

Please sign and return this form by (date) to:

(address)

I acknowledge receipt of the referenced document(s). I understand the document(s) describe quality assurance, quality control, data management and reporting, and other technical activities that must be implemented to ensure the results of work performed will satisfy stated performance criteria.

Signature Date

ATTACHMENT 2 GPS POLICY

Dr. David Weindorf, TSU, will be responsible for GPS training and use. He will follow the following guidelines:

GLOBAL POSITIONING SYSTEM: DEFINITIONS
Datum - A mathematical model used by cartographers to define the shape of the earth in a specific area. Mapping applications in the United States are normally based on either the North American Datum of 1927 (NAD27) or the newer, more accurate North American Datum of 1983 (NAD 83). The coordinates of a given point depend on which datum is used.
Differential Correction - A process applied to raw GPS data that removes certain types of error; primarily, the error introduced by Selective Availability. This process requires correction data from a reference GPS receiver operating from a precisely known location. The process can be performed in "real time" if the reference receiver broadcasts the correction data and if the user can receive the correction data.

GIS - Geographic Information System. A collection of hardware, software, data and procedures to collect, store, manage, query and analyze spatial data as well as traditional tabular data. Computer mapping is a part of GIS, but beyond that, GIS adds a spatial dimension to all types of geography-based tabular data, allowing new, powerful tools for data integration, query and analysis in a wide variety of applications.

GPS - Global Positioning System. A satellite-based system managed by the Department of Defense to allow absolute geographic position measurement worldwide.

GPS Data, Raw - Positional data obtained by a GPS receiver before errors due to Selective Availability have been removed. These positions are typically only accurate to 100 meters.

GPS Data, Corrected - Positional data obtained by a GPS receiver that has been differentially corrected to remove certain types of error, primarily Selective Availability. These positions are considered accurate to within twelve meters or even one meter, depending on equipment and procedures used.

Metadata - Metadata is "data about data." In the case of positional data, the data stored in a database will include not only the latitude and longitude of a location, but also additional data elements that describe how and when the position was measured, and an assessment of the accuracy of the measurement. These additional data elements are called metadata.

Minimum Elevation - A GPS processing parameter which determines how high in the sky a satellite must be in order for the receiver to accept data from it for calculating a position. Measured in degrees of arc starting at the horizon.

PDOP - Positional Dilution of Precision. A measure of the quality of a GPS measurement taken from a given set of four satellites at a given time. If the satellites are not widely distributed from the user's location, the PDOP value will be higher, and the quality of the measurement will be diminished. PDOP values over 6 are generally not acceptable.

SA - A procedure used by the Department of Defense to limit the positional accuracy available to non U.S. military users of GPS. Errors introduced by SA can be effectively removed through differential correction techniques.

8.12.2 GLOBAL POSITIONING SYSTEM: Guidelines and Information

GPS TRAINING

To ensure that the TSSWCB receives reliable and accurate positional data, all project personnel and contractors that will collect positions with GPS must first be trained.

A TSU GPS training program will serve to:

- Make appropriate GPS training easily available.
- Ensure that staff training is sufficient to cover GPS techniques normally used within the Agency.
- Identify each certified individual with a certificate number.
- Provide reasonable validation that the accuracy of positional data obtained through GPS meets the Agency's GIS Positional Data Policy.

TRAINER QUALIFICATION. The GPS trainer must be qualified to give GPS certification training and have actual field GPS data collection experience.

Minimum lecture and/or demonstration elements include:

- Background of the Global Positioning System.
- GPS accuracy issues.
- Operation of GPS equipment, including basic troubleshooting.
- Data collection procedures.
- Differential correction, both real time processing and post processing.
- Coordinate averaging for point locations.
- Data output in formats appropriate for import to GIS or tabular databases.

Minimum hands-on exercises, to be successfully completed by each student, include:

- Pre-planning, including data quality objectives, equipment and materials needed, logistics of field data collection, and prediction of GPS data collection conditions.
- Navigation to a given coordinate.
- Storing and transferring raw positional data.
- Differential correction of raw data through post processing.
- Averaging corrected point data and outputting to a GIS file.

- Class exercises shall also include computer plotting of point data to allow students to better understand GPS accuracy issues and the effects of differential correction and point data averaging.

EQUIPMENT FOR GPS TRAINING. The student and his/her Division are responsible for providing appropriate GPS equipment required for certification training. Although each student is encouraged to use their own unit during the class exercises, the trainer may approve sharing of units, no more than two students to one unit. In the event that units are shared, it is essential that each student directly performs each of the exercise steps at least once.

OFFSET MEASUREMENT. It is sometimes impossible, or impractical, to place a GPS receiver immediately on top of or adjacent to the site being positioned. In this case it is appropriate to obtain a GPS position at a nearby point with a known offset (X/Y or Bearing/Range) from the site. In these cases, potential error associated with the offset measurement must be added to the potential error associated with the GPS measurement in order to assess the accuracy of the site position.

ACCURACY LEVEL. A Program Area may impose higher accuracy requirements for certain purposes, as appropriate. It is the Division's responsibility to develop the proper procedures, including training for specialized techniques such as offset measurements, to ensure that the Division's required accuracy level is obtained. In the event that a position is needed that cannot meet the 25 meter accuracy standard, the position will not be considered certified, and the lower accuracy level will be reflected in the associated metadata.

GPS EQUIPMENT STANDARDS

Many models of GPS receivers and accessories are available from a number of manufacturers. These receivers differ greatly in accuracy and features; some are incapable of making measurements that meet the Agency's horizontal accuracy standard. To ensure that the Agency can get maximum benefit from its investment, the following minimum specifications will apply to all GPS equipment procurements:

GPS RECEIVER. A GPS receiver can be either a standalone unit, or a GPS module plugged into a portable computer. The GPS receiver must:

- Have six channel parallel reception or better.
- Employ these processing parameters:
- Position acquisition rate - 1/second or better
- Position mode - 3D (uses 4 satellites)
- Maximum PDOP - 6 (or less)
- Minimum Elevation - User-selectable*

- *The elevation mask requirement may be waived if the Division can show that post-processing differential correction will never be needed on raw data obtained by the receiver.
- Have the ability to perform real-time differential correction.

- Have the ability to store at least 180 raw position measurements for the purpose of post-processing differential correction. (This requirement may be waived if the Division can show that it will never need to perform post-processing differential correction on raw data obtained by the receiver.)
- Have the ability to transfer almanac and position data to a personal computer via a serial port.
- Include software to perform mission planning, differential correction, point data averaging, and conversion to common formats.
- Have a water and shock resistant case.
- Include portable power source(s) which will last a full working day.

REAL-TIME CORRECTION RECEIVER. This may be a standalone unit, or it may be integrated within the GPS receiver. The real-time correction receiver must:

- Receive correction data from a recognized, reliable source, and which is appropriate for real-time correction in the geographic area in which the GPS measurements will be made.
- Output correction data in RTCM-SC-104 (Radio Technical Commission of Maritime Service - Special Committee Paper No. 104) format via an RS-232 cable that matches GPS receiver.
- Include portable power source(s) which will last a full working day.

GPS DATA COLLECTION STANDARDS

Detailed data collection procedures will be developed at the Division level and will be in accordance to this Policy. Divisions are strongly encouraged to utilize real-time differential correction techniques whenever possible, to reduce staff time and to eliminate the possibility that an error may occur during post-processing differential correction. The Division's procedures should take into the account the possible need for post-processing differential correction in the event that real-time correction fails due to signal loss.

- When using real-time correction, the correction data must be obtained from a recognized, reliable source, as determined by the GPS Coordinator.
- A single position reading obtained through appropriate use of real-time correction will be certified under this Policy as meeting the 25 meter horizontal accuracy standard. However, in the interest of obtaining better accuracy for little cost (about two minutes of staff time), staff is encouraged to average 100 or more positions to arrive at a final measured position.
- When using post-processing correction to measure a point location, staff must store at least 180 uncorrected positions in a file. Correction data must be obtained from a recognized, reliable source (such as the reference network maintained by the Texas Department of Transportation), as determined by the GPS Coordinator. The corrected positions should be averaged to produce a final measured position.
- All GPS measurements should be taken using a set of four satellites which are in a favorable configuration. The Positional Dilution of Precision (PDOP) is a recognized method to quantify how well the satellites are configured. The GPS receiver must be set to not record positions during times that the PDOP exceeds a value of 6.

MINIMUM DATA ELEMENTS

All site positions measured with GPS and stored in a project database or spreadsheet file will include, at a minimum, the following data elements:

- Latitude – in decimal degrees, using NAD83 datum.
- Longitude – in decimal degrees, using NAD83 datum.
- Method of Collection – using standard EPA codes.
- Date of Collection – date the GPS measurement was taken.
- Horizontal Accuracy Assessment – value in meters.
- GPS Certificate Number – to identify who made the measurement.

DATA DICTIONARY. The Steering Committee will produce a data dictionary to aid in implementing these data elements in all databases that receive GPS positions.

GEOGRAPHIC IDENTIFICATION NUMBER. An additional data element, called a Geographic Identification Number, is required under GIS Positional Data Policy when positions are entered into a Geographic Information System.

ATTACHMENT 3: Soil Sampling and Analysis Procedures, Tarleton State University, Dr. David Weindorf

The following procedures are taken from:

Pierzynski, G.M. (Ed). 2000. Methods of Phosphorous Analysis for Soils, Sediments, Residuals and Waters. Southern Cooperative Series Bulletin #396.

Sampling Procedures:

The collection of a representative and reliable soil sample for phosphorus (P) analysis requires predetermination of sampling depth, position relative to nutrient application patterns, and sampling intensity within the field. The appropriate soil sampling depth is dependent upon the planned interpretation of the analytical data. If investigation of P distribution or concentration with depth is a specified research objective, three factors must be considered when determining the appropriate sampling depth: 1) influence of changes in soil morphology with depth (i.e., horizonation); 2) influence of surface soil management (e.g., tillage); and 3) necessity to maintain sample collection depth uniformity across numerous sites.

Sample collection depth based on observed morphological horizon depths is quite useful when attempting to associate soil P measurements with soil physical properties. This technique may generate very reliable data for a particular, well-defined location, but this laborious task is not very practical when a research project focuses on more than a few soils or when the data will be subjected to broader, perhaps watershed-scale, interpretation.

Depth of tillage will dramatically impact soil P distribution with depth. Tillage depth is seldom constant across a given field. Sampling depths should include soil collected from a depth confidently within the tillage zone and excluding soil from below the tillage zone. A second transitional depth should be collected that is expected to be variably affected by tillage and includes the lower tillage boundary. Deeper sampling depths should not be directly impacted by physical tillage activity.

Relating soil physical and chemical properties to the potential for P transport with surface runoff water requires a different approach to soil sample collection. Sharpley (1985) studied five soils of varying physical and chemical properties and found that effective depth of interaction between surface soil and runoff ranged from 2 to 40 mm. The effective depth of interaction varied by soil type, surface slope, rainfall intensity, and crop residue. For most agricultural soils, samples collected to a depth of 20 mm would accurately define the effective depth of runoff interaction generated by moderate to high rainfall intensity (< 50 mm/h). For medium to coarse textured soils on steeper slopes (>12 %) that are subjected to high intensity rainfall (> 100 mm /h), soils should be sampled to a depth of 40 mm in order to more accurately relate the potential for P transport with surface runoff to soil physical and chemical properties.

Recommended soil sampling intensity is usually between 10 and 30 subsamples per composite sample (Whitney et al., 1985; Kitchen et al., 1990; Coale, 1997). A single composite sample may represent a single research plot or an entire production field, but generally not more than 10 ha.

Discrete nutrient application patterns in a field can increase the complexity of appropriate soil sample collection procedures. In a review of positional P availability resulting from band application of fertilizer P, Sharpley and Halvorson (1994) stated that collection of 15 random samples (Ward and Leikam, 1986; Shapiro, 1988) to 30 random samples (Hooker, 1976) were adequate to reflect crop P availability in conventionally Methods for P Analysis, G.M. Pierzynski (ed) tilled fields where previous P fertilizer bands exist. For no-till or minimum-till soils containing residual P fertilizer bands in which the location of the P bands is known, sampling to include one "in-the-band" soil sample for every 20 "between-the-band" samples for 76 cm band spacing, and one "in-the-band" sample for every 8 "between-the band" samples for 30 cm band spacing, will accurately reflect the mean soil P status of the field (Kitchen et al., 1990). Twenty to 30 subsamples per composite are adequate. When the location of the P bands is not known, collection of 20 to 30 subsamples per composite is also adequate but paired subsamples should be collected where the location of the first subsample of the pair is completely random and the second subsample of the pair is located 50% of the band-spacing distance from the first, perpendicular to the band direction (Kitchen et al., 1990).

Sample Handling, and Preparation and Storage:
Air-drying should be satisfactory for investigations into relative changes in soil P concentrations in response to imposed treatments or for routine comparative P analyses. Soil samples should be air-dried (25 to 30°C) and crushed to pass a 2 mm sieve. Air dried and crushed soil samples are stable at room temperature. Air-drying may not be suitable for determination of the absolute quantity of the various P fractions in soils. Air drying may artificially elevate the quantity of soluble reactive P above *in situ* conditions. Bartlett and James (1980) studied P solubility in the surface soil of a loamy fine sand and found water-soluble P concentrations to be five times higher in air-dried samples (~30 mg P/L) than in samples stored at field moisture (~5 mg P/L). The effect of air-drying was only partially reversed by rewetting and incubating the air-dried soil for one month (~20 mg P/L). Water-soluble P in rewetted soil samples that had previously been air dried was shown to decrease during three months of storage at 20°C (Bartlett and James, 1980). For quantitative characterization studies, soil and sediment samples should be stored at field moisture content under refrigeration, between 0 and 4°C. Soil and sediment samples should not be stored frozen (<0°C), because the water-soluble proportion of total P increases after freezing (Mack and Barber, 1960). Mixing moist soil samples to achieve homogeneity is difficult, and careful attention should be paid to ensure thorough mixing prior to subsampling. Moist soils are also difficult to sieve, but large particles (> 2mm) should be removed from the sample prior to analysis.

References:
Bartlett, R. and B. James. 1980. Studying dried, stored soil samples - some pitfalls. Soil Sci. Soc. Am. J. 44:721-724.

Coale, F. J. (ed.) 1997. Chesapeake Bay region nutrient management training manual. USEPA Chesapeake Bay Program, Annapolis, MD.

Hooker, M.L. 1976. Soil sampling intensities required to estimate available N and P in five Nebraska soil types. MS Thesis, Univ. Nebraska, Lincoln, NE.

Kitchen, N.R., J.L. Havlin, and D.G. Westfall. 1990 Soil sampling under no-till banded phosphorus. Soil Sci. Soc. Am. J. 54:1661-1995. Methods for P Analysis, G.M. Pierzynski (ed)

Mack, A.R. and S.A. Barber. 1960. Influence of temperature and moisture on soil phosphorus. I. Effect on soil phosphorus fractions. Soil Sci. Soc. Am. Proc. 24:381- 385.

Shapiro, C.A. 1988. Soil sampling fields with a history of fertilizer bands. Soil Sci. News, Nebraska Coop. Ext. Serv., Vol. 10, No. 5.

Sharpley, A.N. 1985. Depth of surface soil-runoff interaction as affected by rainfall, soil slope, and management. Soil Sci. Soc. Am. J. 49:1010-1015.

Sharpley, A.N. and A.D. Halvorson. 1994. The management of soil phosphorus availability and its impact on surface water quality. p. 7-90. *In* R. Lal and B. A. Stewart (ed.) Soil processes and water quality. Advances in Soil Science. Lewis Publishers, Boca Raton, FL.

Ward, R. and D.F. Leikman. 1986. Soil sampling techniques for reduced tillage and band fertilizer application. *In* Proc. Great Plains Soil Fertility Workshop. March 4-5,1986. Denver, CO.

Whitney, D.A., J.T. Cope, and L.F. Welch. 1985. Prescribing soil and crop nutrient needs. p. 25-52. *In* O. P. Engelstad (ed.) Fertilizer technology and use. 3rd ed. SSSA, Madison, WI.

TP Procedure (Mehlich III):

Introduction:

The Mehlich 3 soil test was developed by Mehlich in 1984 as an improved multielement extractant for P, K, Ca, Mn, Cu, Fe, Mn, and Zn (Mehlich, 1984). Today, the Mehlich 3 test is used throughout the United States and Canada because it is well suited to a wide range of soils, both acidic and basic in reaction. The Mehlich 3 extractant was selected by workers in the southern region as the standard reference procedure for soil test P determination (Tucker, 1992). The Mehlich 3 is similar in principle to the Bray and Kurtz P-1 test because it is an acidic solution that contains ammonium fluoride. Acetic acid in the extractant also contributes to the release of available P in most soils. It is more effective than the Mehlich 1 soil test at predicting crop response to P on neutral and alkaline soils because the acidity of the extractant is neutralized less by soil carbonates (Tran and Simard, 1993). Several studies showed that the Mehlich 3 soil test is highly correlated with P extracted from soils by the Bray and Kurtz P-1, Mehlich 1, and Olsen P methods (Sims, 1989; Tran et al., 1990; Wolf and Baker, 1985).

A Mehlich 3 value of 45-50 mg P/kg soil is generally considered to be optimum for plant growth and crop yields, higher than the critical values used for other standard soil P tests such as the Bray and Kurtz P-1, Mehlich 1, and Olsen P.

Equipment:

1. No. 10 (2 mm opening) sieve
2. Standard 1 cm^3 , 2 cm^3 (or 2.5 cm^3) stainless steel soil scoops
3. Automatic extractant dispenser, 25 mL capacity
4. Extraction vessels, such as 50 mL Erlenmeyer flasks, and filter funnels (9 and 11 cm) and racks
5. Rotating or reciprocating shaker with a capability of 200 excursions per minute (epm)
6. Whatman No. 42 or No. 2 (or equivalent) filter paper, 9 to 11 cm. (Acid resistant filter paper may be needed if using an automated method for determining P concentration by intensity of color. Bits of filter paper may cause an obstruction in the injection valves.)

Reagents:

1. Mehlich 3 Extracting Solution: (0.2 M CH_3COOH, 0.25 M NH_4NO_3, 0.015 M NH_4F, 0.013 M HNO_3, 0.001 M EDTA [$(HOOCCH_2)_2NCH_2CH_2N$ $(CH_2COOH)_2$]. Prepare as follows:
   Ammonium fluoride (NH4F) and EDTA stock solution (3.75 M NH_4F:0.25 M EDTA)
2. Add 1,200 mL of distilled water to a 2 L volumetric flask.

3. Add 277.8 g of NH_4F and mix well.
4. Add 146.1 g EDTA to the solution.
5. Make solution to 2 L, mix well and store in plastic (stock solution for 10,000 samples).

Mehlich 3 extractant preparation

6. Add 8 L of distilled water to a 10 L carboy.
7. Dissolve 200 g of ammonium nitrate (NH_4NO_3) in the distilled water.
8. Add 40 mL NH_4F-EDTA stock solution and mix well.
9. Add 115 mL glacial acetic acid (99.5%, 17.4 M).
10. Add 8.2 mL of concentrated nitric acid (HNO_3, 68 to 70 %, 15.5 M).
11. Add distilled water to 10 L final volume and mix well (enough extractant for 400 samples), final pH should be 2.5 ± 0.1.

Procedure:

1. Scoop or weigh 2.0 g of soil into a 50 mL Erlenmeyer flask, tapping the scoop on the funnel or flask to remove all of the soil from the scoop. Where disturbed bulk density of soil varies significantly from 1.0 g cm_3, record both weight and volume of samples. (Standard 2.5 cm_3 scoops may also be used, but a 1:10 soil:extractant volumetric ratio should be maintained)
2. Add 20 mL of extracting solution to each flask and shake at 200 or more epm for five minutes at a room temperature at 24 to 27°C.
3. If it is necessary to obtain a colorless filtrate, add 1 cm_3 (~200 mg) of charcoal (DARCO G60, J. T. Baker, Phillipburg, NJ) to each flask.
4. Filter extracts through Whatman No. 42 filter paper or through a similar grade of paper. Refilter if extracts are not clear.
5. Analyze for P by colorimetry or inductively coupled plasma emission spectroscopy using a blank and standards prepared in the Mehlich 3 extracting solution.

Calculations:

Mehlich 3 Extractable P (mg P/kg) = [Concentration of P in Mehlich 3 extract, mg P/L] x [0.020 L extract ÷ 0.002 kg soil]

References:

Mehlich, A. 1984. Mehlich 3 soil test extractant: A modification of the Mehlich 2 extractant. Commun. Soil Sci. Plant Anal. 15:1409-1416.

Tucker, M.R., 1992. Determination of phosphorus by Mehlich 3 extractant. *In* Donohue, S.J. (ed.) Reference Soil and Media Diagnostic procedure for the southern region of the United States. So. Coop. Series Bulletin 374. Va. Agric. Exp. Station, Blacksburg, VA. p. 9-12.

Sims, J. T. 1989. Comparison of Mehlich 1 and Mehlich 3 extractants for P, K, Ca, Mg, Mn, Cu, and Zn in Atlantic Coastal Plain soils. Commun. Soil Sci. Plant Anal. 20:1707-1726.

Tran, T. Sen and R.R. Simard. 1993. Mehlich 3 extractable elements. p. 43-49. *In* M.R. Carter (ed.) Soil Sampling and Methods of Analysis. Can. Soc. Soil Sci., Ottawa, Ontario.

Tran, T. Sen, M. Giroux, J. Guilbeault, and P. Audesse. 1990. Evaluation of Mehlich 3 extractant to estimate available P in Quebec soils. Commun. Soil Sci. Plant Anal. 21:1-28.

Wolf, A.M. and D.E. Baker. 1985. Comparison of soil test phosphorus by the Olsen, Bray P1, Mehlich 1 and Mehlich 3 methods. Commun. Soil Sci. Plant Anal. 16:467-484.